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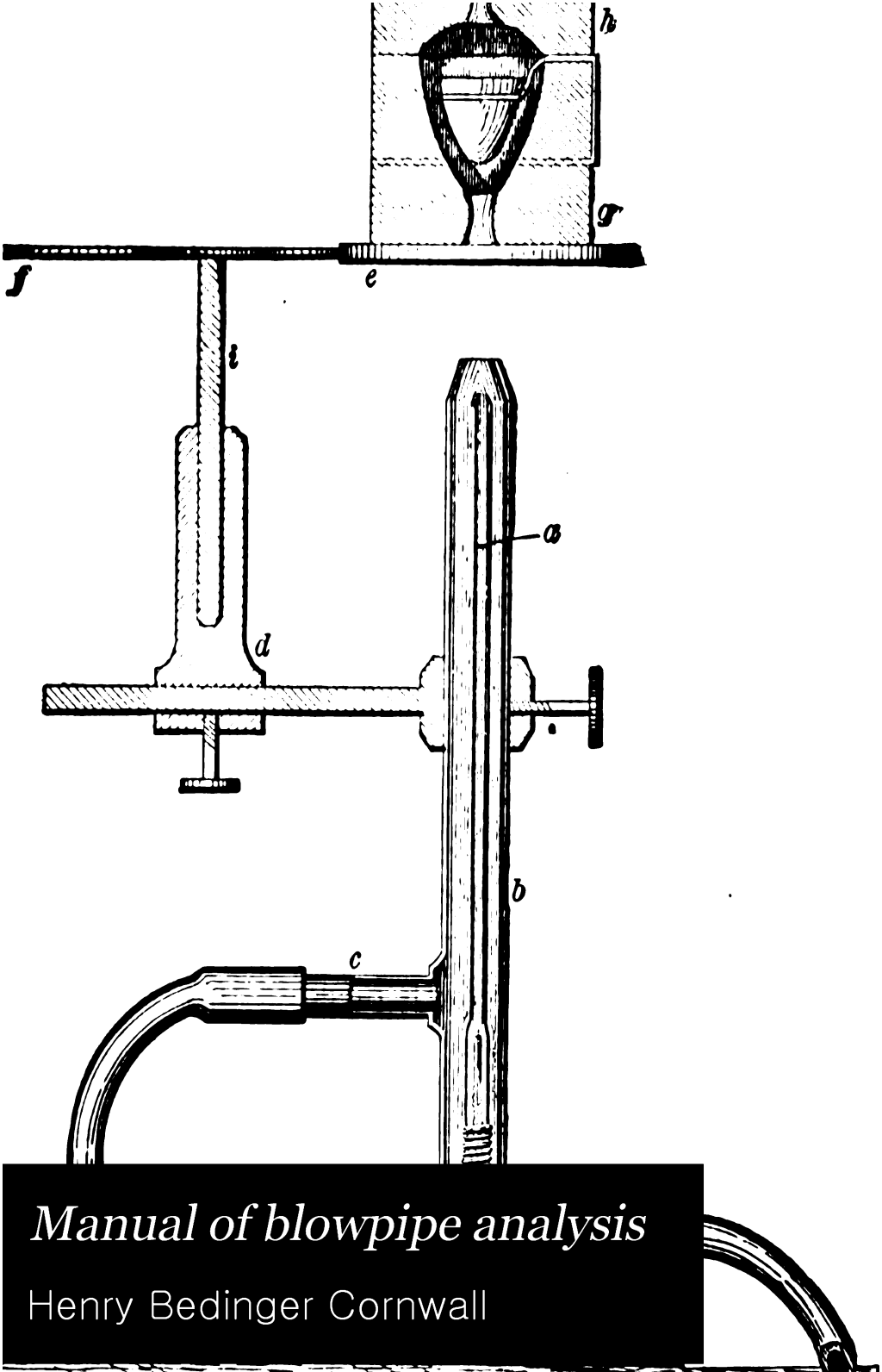
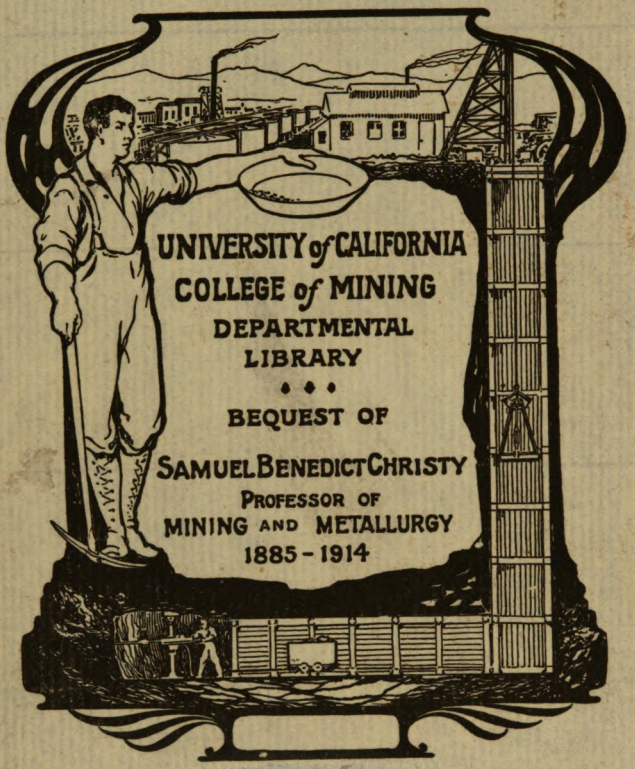


Manual of blowpipe analysis

Henry Bedinger Cornwall



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S. B. CHRISTY.

MANUAL

OF

BLOWPIPE ANALYSIS,

QUALITATIVE AND QUANTITATIVE,

WITH

A COMPLETE SYSTEM

OF

DETERMINATIVE MINERALOGY.

BY

H. B. CORNWALL,

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WITH SIXTY-NINE WOOD-CUTS AND ONE LITHOGRAPH PLATE.

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PREFACE.

After a somewhat extended experience in teaching the use of the blowpipe, the author has prepared this manual with the desire to furnish for students a complete guide in all branches of blowpipe analysis.

He desires freely to acknowledge his indebtedness to Plattner's well-known work, without the aid of which, indeed, no really useful manual of blowpipe analysis could be produced. In the preparation of Chapters III. and VII. especially, aid has been obtained both from the third American edition of Plattner and from the fifth German edition, Leipsic, 1878. The chapter on quantitative analysis contains a concise description of Plattner's methods, with the addition of certain details in regard to the operations, as well as descriptions of a few pieces of serviceable automatic apparatus, while the figures of instruments have also for the most part been taken from Plattner, since they represent the best forms of apparatus.

The manual being intended as well for students working alone as for use with classes, contains some details and repetitions which may seem unnecessary to the trained chemist, but it is believed that these will tend also to lessen the labors of the instructor. The chapter on systematic analysis has been prepared with the hope that it will prove useful by giving to the beginner an insight into the methods to be followed in the examination of complex substances, and especially of ores and metallurgical products.

It has also been deemed advisable to devote a chapter to the characteristics of important ores, particularly their blowpipe characteristics, while a system of determinative mineralogy has been introduced, both because the blowpipe renders such great service in the examination of minerals, and because the determination of minerals affords excellent practice in qualitative blowpipe analysis. The system has been devised with the view of employing the blowpipe to the exclusion, as far as possible, of wet methods, and although it is true that in certain cases it requires a somewhat greater experience in blowpipe analysis, it is believed that this experience can be readily acquired. A part of the system, pages 283 to 291, including many

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minerals which can be determined only in part by the blowpipe, has been taken with little change from von Kobell's *Tafeln zur Bestimmung der Mineralien*, 11th ed., Munich, 1878; pages 260 to 264 have also been adapted in part from the *Tafeln*, but otherwise the arrangement of the system is for the most part new. The details of some of the tests have also been taken from von Kobell, while Plattner's work and Naumann's *Elemente der Mineralogie*, 10th ed. (Zirkel's), Leipsic, 1877, have also been consulted in the preparation of Chapters VIII. and IX., but the blowpipe behavior of nearly all of the commoner minerals and of a large number of the rarer ones, has been especially studied for the same purpose. Fifty or sixty of the minerals included in pages 283 to 291 have been arranged in a separate sub-section, pages 291 to 296, in order that the student might more rapidly learn the methods to be used in their determination, and at the same time become acquainted with the behavior of the minerals themselves, which include some of the most important silicates of the alkalies and earths. In the method of writing the mineral formulas, which are mainly taken from the fifth German edition of Plattner, the author has followed the examples of Plattner and von Kobell. An explanation of the formulas is given in Chapter IX.

A few blowpipe tests which are either new or modifications of already known tests, have been introduced, some of which will be found in Chapter III., under Potassium, Titanium, Columbium, Lead, Cadmium, Molybdenum, Tungsten and Iodine.

In addition to the works already cited, aid has been occasionally obtained from Kerl's *Leitfaden bei Löthrohruntersuchungen*, Clausthal, 1877; Fresenius's *Manual of Qualitative Chemical Analysis*, Amer. ed.; J. D. Dana's *System of Mineralogy*; and G. J. Brush's *Determinative Mineralogy and Blowpipe*, which work contains a very complete tabular arrangement, not only of the chemical, but also of the physical characteristics of minerals, of great use to the student of mineralogy.

HENRY B. CORNWALL.

JOHN C. GREEN SCHOOL OF SCIENCE,
Princeton, N. J., *August*, 1882.

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INTRODUCTION.

A. DIRECTIONS FOR SELF-INSTRUCTION.

THE student who may be working alone will perhaps find the following outline of a short course in qualitative blowpipe analysis of use to him. It is supposed that he is at least acquainted with the elements of inorganic chemistry.

After reading about the use of apparatus, coals, glass tubes, etc., and learning to manage the flame, testing his proficiency in that respect as directed in §§ 25 to 28, he should study the general operations and examinations of qualitative analysis, especially those indicated below, taking as examples the substances indicated by the numbers* in square brackets [ ] in the text referred to, and reading carefully all the remarks bearing upon the operations in hand. If he desires to prepare himself especially for the determination of minerals he may perform the following tests.

Examination in the closed tube: §§ 30, *d;* 31, 2, *a, b, c, d.*

Examination in the open tube: § 32, 1, *a, b;* 2, *a, b;* 3, *b.*

Examination on charcoal alone (using both the metals and their oxides, when examples of both are cited): §§ 33, *a, b, g;* 35, *a, b, c, d, e, g;* 37, *a, b;* 38, *a, b, c.*

All the examples cited in the examinations for *fusibility*, § 40; *alkalinity*, § 41; *flame-coloration*, § 42; practice in *roasting*, § 43, using [15] or chalcopyrite, see list of minerals, p. 254; tests with the *glass fluxes*, reading §§ 44 to 46, practicing the reduction, § 44, 1, *d*, on lead and copper oxides there described, and then making the tests for the commoner oxides, § 46, trying afterwards unknown simple oxides by Table I., § 47. It will be well also to study the colors produced in the glass fluxes by a few oxides mixed in varying proportions, using Tables II. to V. as a guide and trying especially (with salt of phosphorus as well as with borax) mixed oxides of the following metals:—iron and manganese; iron and cobalt; iron and copper; cobalt and manganese; cobalt and nickel; nickel and copper.

Reduction with soda: §§ 50 to 52, especially practicing on tin oxide § 51, washing the resulting metal in the agate mortar; also preparing pure copper from chalcocite, and silver from pyrargyrite or argentite by the reduction with soda, followed by purification with borax, p. 68.

---

* These numbers refer to the list, p. 3.

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Formation of a coat with soda on coal, § 53, using [11], or sphalerite.
Fusibility of substances with soda: § 54, trying [27] and [51].
Examination with cobalt solution: § 55.

The following tests should then be made, among those in Chapter III., intended for special cases when the presence of other disturbing substances must be considered: § 56, foot-note, p. 93, using [46]; §§ 60; 67, *b;* 69, *d;* 72, *c;* 76, *d*, using a suitable specimen of niccolite, smaltite, etc., if a single mineral can be found containing arsenides of iron, cobalt and nickel in sufficient quantity; otherwise an artificial arsenide [64] may be prepared from mixed metallic oxides by arsenicizing them, § 185; or else separate trials may be made on arsenopyrite, smaltite and niccolite, to obtain the characteristic reactions for iron, cobalt and nickel respectively, with perhaps slight reactions for one or both of the other metals at the same time.

The special tests will then be continued on the metals, studying the properties of the metals obtained from the oxides by reduction tests, in so far, at least, as that may not have been already done: §§ 83, *a*, *b* (using for this last test a mixture of oxides of silver and copper); 84, *a;* 85; 86, *a*, *b;* 87; 88, *a;* 88, *c* (a reduction with lead, followed by separation with boric acid, as in *f*, 2); 89; 90, *b;* 92, using pure gold and also an alloy with silver, to be separated by nitric acid; 94; 95, especially the green flame-coloration from the coat; 97, *a*, especially the volatility of the coat and the alliaceous odor; 99, *f;* 100, *d;* 101, *d;* 103, *b*, both the sulphur dioxide odor on coal in O. F. [15] and the test in the open tube, using blue litmus paper; *c*, reduction (with soda alone) using any sulphate; *h*, use [51] for both; *i;* 104, *a*, 1, use [26]; 107, *b*, *d*, use [4]; 109, *b*, *c*, use [53] for both; 110, *a* (deflagration) use potassium nitrate; 111, *a* [27]; 112, *a* (formation of a silica skeleton), *c*, use [44] for both and under *c* fuse with soda; *d*, try all the minerals mentioned, as the action of acids requires careful study (see also p. 252); 113, *a* (using Turner's flux); *d*, 1, using borax; 2, on [66] after fusion with soda.

The student having completed these tests carefully will be ready to proceed to Chapter IX.

When a course preparatory especially to the examination of metallurgical products and the full analysis of ores, etc., is desired, the student, after completing the outline given above (of which some parts referring to lithium, zirconium, nitrates, and similar substances not ordinarily of interest to the smelter, may be omitted) should turn to §§ 116–123 and work up the examples there given, either by means of real products, or alloys and mixtures representing them. The alloys are in general easily made by fusing together the necessary ingredients on coal in the reducing flame, adding a little borax to keep them bright. He can then refer to the scheme, p. 150, and ascertain from it what special tests of Chapter III. need to be studied and can practice them by means of suitable test substances, readily prepared from the materials in the list already cited. A similar course, together with the more extended study of the special tests in Chapter III., is recommended for the chemist.

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B. LIST OF SUBSTANCES FOR PRACTICE.

(For full list of minerals see p. 254).

1. Molybdic acid.
2. Manganese binoxide.
3. Copper oxide.
4. Fluorite.
5. Zinc oxide.
6. Cerussite.
7. Malachite.
8. Siderite.
9. Quill clippings.
10. Lead nitrate.
11. Calamine.
12. Zircon.
13. Mercurous chloride.
14. Arsenous oxide.
15. Pyrite.
16. Realgar.
17. Stibnite.
18. Cinnabar.
19. Tiemannite.
20. Arsenic.
21. Smaltite.
22. Arsenopyrite.
23. Mercuric oxide.
24. Cadmium.
25. Cadmium oxide.
26. Sodium chloride (common salt).
27. Calcite.
28. Zinc oxide.
29. Antimony.
30. Antimonous oxide.
31. Zinc.
32. Tin.
33. Tin binoxide.
34. Lead.
35. Bismuth.
36. Bismuth trioxide.
37. Silver.
38. Silver oxide.
39. Cupric chloride.
40. Prehnite.
41. Strontianite.
42. Vermiculite, or Pyrophyllite.
43. Natrolite.
44. Almandite.
45. Actinolite.
46. Orthoclase.
47. Bronzite.
48. Gypsum.
49. Potassium carbonate.
50. Lepidolite.
51. Barite.
52. Boric acid.
53. Apatite.
54. Quartz.
55. Tungstic acid.
56. Rutile.
57. Hematite.
58. Uranic oxide.
59. Chromic oxide.
60. Cobalt oxide.
61. Nickel oxide.
62. Magnesite.
63. Alum.
64. Speiss.*
65. Wollastonite.
66. Tourmaline.

C. SPECIAL LIST OF APPARATUS FOR QUALITATIVE BLOWPIPE ANALYSIS.

The following limited list of apparatus, for each student, will be found useful in a course of blowpipe analysis for the simpler tests and for determinative mineralogy.

Bunsen burner,† low form with cap ; blowpipe, Freiberg pattern,‡ or Black's, § 1 ; alcohol lamp (not needed if the Bunsen burner is employed) ; matrass holder ; platinum wire holder and wires ; platinum forceps, § 10, *a*, Fig. 11 ; steel forceps (only when oil is used), Fig. 13 ; hammer ; anvil ; spatula coal borer, Fig. 14 ; dirt tray ; scissors (only for the oil lamp) ; knife ; magnifying glass, § 10, *i* ; magnet, § 10, *v* ; ounce bottles for soda, salt of phosphorus and borax ; two small porcelain dishes ; watch glasses ; cobalt glass.

---

* Arsenide of iron, cobalt, nickel and copper (artificial).

† If gas cannot be used the oil lamp, § 2, is the best substitute, and the oil lamp *must be used* for a full course in qualitative blowpipe analysis.

‡ The Freiberg pattern is almost indispensable for a full course, when oil is to be used.

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Agate mortars and the reagents more frequently needed, except the three above named, may be used by several students in common. If gas is used one or two oil lamps will generally be needed for the class to use in testing for sulphur by reduction with soda. Several water baths, test-tubes, and similar accessory apparatus are also to be provided for general use.

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CHAPTER I.

APPARATUS, REAGENTS AND OPERATIONS.

I. Blowpipe and Lamp.

1. *The Blowpipe.* (The letters B.B. mean "before the blowpipe.") The best general form, one necessary for the most complete work in every branch of blowpipe analysis, is the Freiberg model (Lingke's) of Berzelius's blowpipe, shown in somewhat less than one-half its true size in Fig. 1. It consists of a brass, or still better, German silver tube *AB*; a trumpet mouth-piece *C*, of horn or hard rubber, carefully shaped, with a proper flare, so as to set well against the lips without hurting them, and from 38 to 40 millim. wide; a chamber *A*, to retain the moisture condensed from the breath; and a short tube *a*, carrying the platinum jet *b*. The entire length of the blowpipe may be two hundred and thirty millim. or a little more or less, as suits the operator.

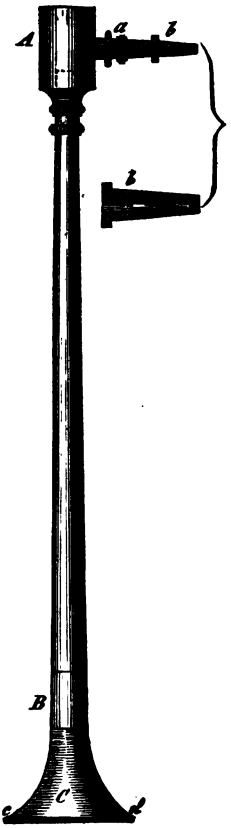
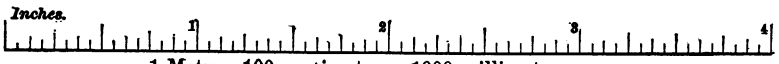


Fig. 1.

No part of the ordinary qualitative apparatus requires to be more carefully made than the platinum jet, but many dealers are very careless in this respect. The jet is often too thin to endure any but the most careful handling, the aperture in it is made on one side instead of in the center, or is so ill-shaped as to give a bad flame, and is rarely made large enough, although this last is a defect easily remedied by practiced hands. In determining minerals especially it is of the utmost importance that the jet should furnish a steady, well-shaped inner flame, § 26,



1 Metre=100 centimetres=1000 millimetres.
1 Inch=25.399 millim. 1 Millim=0.03937 inch.

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et seq., which does not flicker even under a powerful blast, because the fusibility of many minerals cannot be tested without such a flame, sufficiently hot to melt the thinnest platinum wire, p. 32, if need be. Too little attention is also given to the shape and size of the trumpet mouthpiece.

The platinum jet, shown in section in about the right full size, is best made of a solid piece ; tips of soldered sheet platinum are less durable. The orifice through which the blast issues from the jet should be, according to Plattner, 0.4 millim. in diameter for general qualitative work and 0.5 for all quantitative work, but a single tip of 0.48 to 0.5 millim. will serve very well for both qualitative and quantitative assays. The taper of the conical tube *a* should correspond to that of the jet, and if then the jet does not furnish a clean, steady flame, even with a very strong blast, it may be that its orifice is too small, or is not circular and smooth. To remedy this defect the orifice is carefully bored out with a slender, triangular steel reamer, or a needle filed to a triangular shape, introduced from the inside of the jet, and the burr thus produced is removed by a fine file. If the hole is too large the jet will be nearly useless. Sometimes the flame is bad because the orifice of the conical tube *a* is too small, and this must then be enlarged by filing off the end. Both jet and tube need occasional cleaning, although very rarely if gas alone is used as a fuel. This is done simply by wiping the tube, inside and out, but the soot or oil is removed from the jet by holding it on a platinum wire, or in the platinum forceps, § 10 *a, just above* the flame of the alcohol lamp or Bunsen burner, until it is burned clean, taking care when using the Bunsen burner, not to heat the jet too highly, if it has a soldered seam.* Solid obstructions can often be removed by blowing through the small end of the jet, or by carefully passing a bristle or fine splinter of wood through the orifice from within. If properly handled a platinum jet will endure many years of constant use.

Cheaper blowpipes, with movable brass jets, serve very well for use with gas, or for any qualitative work. One of the best forms is known as Black's pattern and is made of lacquered tin, or brass, with a movable brass jet. It would be greatly improved if it had a trumpet mouth-piece, which is not usually furnished with these cheaper

* Splits in such seams can be closed by hammering gently, so as to bring the edges together, laying a little fine gold filings and very little powdered borax glass, moistened with as little water as may be, along the well-cleaned platinum, and melting the gold into the seam cautiously, B.B. Such jets are still useful, but require a little more care in burning off soot, etc.

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instruments, but is almost indispensable when a strong blast is to be continued for some time, as in difficult reductions, since the lip muscles become fatigued and cannot keep the lips tightly closed around the tube. All jets except platinum are likely to become corroded and brass jets will often give a color to the blowpipe flame on this account, which the student must not allow to mislead him. The cheaper blowpipes are not quite so well adapted to use with oil or candles, but serve very well with gas, which does not soil them so rapidly. Obstructions are removed from the brass jets by means of a fine splinter of wood. Whatever form of blowpipe is used, it must have a detached jet and a moisture chamber. For ordinary qualitative work and determinative mineralogy there is no objection to the careful use of the cheaper blowpipes. Automatic apparatus, § 147, *g*, is not good for qualitative work.

2. *The Blowpipe Lamp.* For the simpler tests of qualitative chemical analysis and for ordinary purposes of determinative mineralogy the cleanly gas lamps, § 3, are very well suited, and the student who has learned to work with gas can, with a little practice in managing the wick and flame, work even better with the oil lamp; but those who desire to do all that can be done with the blowpipe in the analysis of complicated substances must use the oil lamp, which is also indispensable for all quantitative work requiring a reducing flame. Lamps have been devised in which paraffin is used, the paraffin being run into the lamp while melted, and requiring only to be melted again by the heat from the lamp wick itself before use, and such lamps may sometimes prove

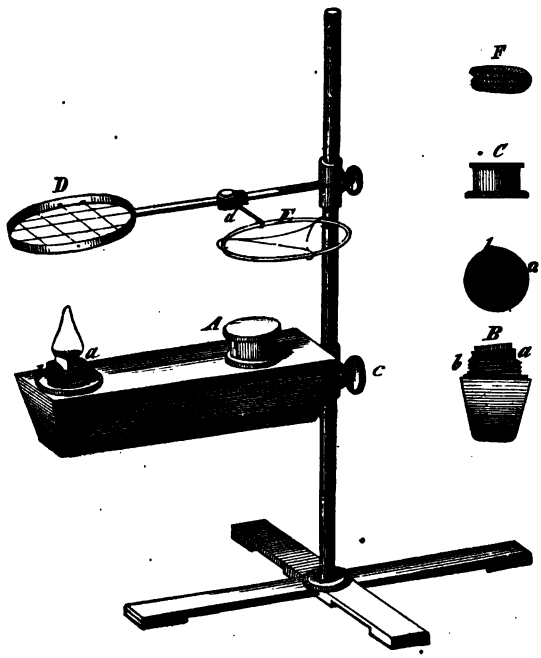

Fig. 2.

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convenient, but the best form of lamp for all general use and the only trustworthy one for quantitative work is that of Berzelius, Fig. 2. It consists of a reservoir for the oil, about one hundred and eight millim. long, a front view of which, about one-third of the natural size, is shown at *B*. This reservoir may be clamped at any height on the stand by the screw *c*, and, as now generally made, has also a swivel joint between the reservoir and the stand, allowing of motion around its horizontal axis, so that the flame may be directed downward at a greater angle than the mere inclination of the top of the wick socket, *a*, would admit of. The reservoir has two caps, *C*, one screwing over the opening *A*, for pouring in oil; the other over the wick socket. This socket, *a b*, should have a clear, rectangular space for the wick to pass through, twelve millim. long (measured on the lower, straight edge) and five millim. wide, since these are the dimensions that best admit the right wick, without pressing too tightly on it. The upper end of the wick socket is filed slanting downward from right to left, so that the flame can be directed downward when needed. Cylindrical cotton wick, as for medium sized argand burners, soft and close woven (not harsh, coarse wick, on which a charred crust quickly forms) is used, folded once lengthwise, making four thicknesses, *F*. Pass it through the socket with the folds opening toward the left, that is, away from the blowpipe jet, and after it has burned a short time trim it quite smooth and parallel to the slanting edge of the wick socket, which should be placed in the lamp so as to slant down toward the left, unless for left-handed persons. The wick must not be too large, or the oil will not rise freely through it, and it should be frequently pressed together with the steel forceps, § 10, *c*, to secure a solid flame. It is better not to trim off all of the charred portion, since only the sharpest scissors will trim the uncharred cotton evenly. To raise the wick, insert one blade of the forceps between the wick and the socket and pry up the former.

The upright rod is screwed into the two cross pieces at the base, and also carries, when needed, an arm terminating in a metal ring, *D*, with a wire net-work or grating, to support larger vessels over the alcohol flame, while a smaller arm carrying a ring, *E*, with a platinum wire triangle, is inserted into a little hole at *d*, and supports small crucibles or vessels that are to be highly heated. A ring to support funnels can also be attached to the lamp stand.

II. Fuel, Gas Lamps, Alcohol Lamp.

3. *Refined rape-seed oil* gives the best flame, of great heating and reducing power, especially necessary for quantitative work. Olive

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oil, or a mixture of twelve parts alcohol and one part turpentine, may be used for quantitative work and they do very well for qualitative tests.

Candles with large wicks will serve for the simpler operations of qualitative analysis and even for determinative mineralogy, if small quantities of substances are used, but do not yield heat enough for many purposes. The wick should be bent in the direction of the blast.

The pure *alcohol flame* is neat and cleanly for heating glass tubes and small vessels, as in evaporations, etc., and is well adapted to observing colored flames, § 42.

Unless a strong reducing action is wanted, *gas* is an excellent fuel. It serves well for the simpler qualitative tests and for determinative mineralogy, but is not to be recommended for all blowpipe work, even in qualitative tests. The most convenient gas-lamp is the low form of Bunsen's burner, now generally to be obtained of dealers in chemical apparatus. It is shown in Fig. 3, *A*, in about one-fourth the true size. The gas enters through rubber tubing attached to the end *a*, and issues from a small orifice at the end of a short tube just within the main tube, about at *b*. Here the gas mingles with air which enters the main tube through a hole in the lower side of that tube at *b*, not shown in the figure (which represents the hole as closed by the ring *d*) and the mixture of air and gas issuing at *c* burns with a feebly luminous, bluish flame, which is of comparatively high temperature and does not deposit soot on bodies held in it. In this respect it resembles the alcohol flame, which it replaces with great advantage for ordinary heating purposes in the laboratory, boiling liquids in glass or porcelain vessels, igniting substances in crucibles, etc.

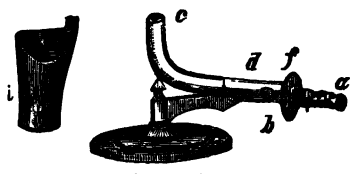

Fig 3. *A*.

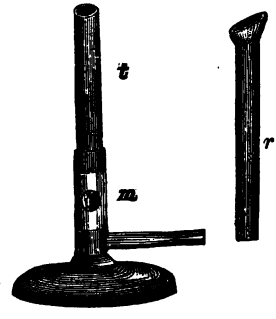

Fig. 3. *B*.

When used for these purposes the short tube, or ring *d*, which encases a part of the main tube and can be turned around that tube by the milled head *f*, is so turned that the hole shown in it in the figure is brought to the lower side of the main tube, allowing air to enter the main tube and mingle with the gas as just described. If, however, the ring *d* is turned so that the hole is in the position shown in

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the figure, no air can enter the main tube at *b*, and the gas alone issuing from *c* burns with its usual yellow, highly luminous flame.

If the cap *i*, about one-third the true size (devised by Prof. A. R. Leeds), is put over the end *c*, the gas flame assumes a flattened form, much like the flame from the flat wick of the oil lamp, and is well adapted to nearly all ordinary blowpipe examinations, with the limitations elsewhere enumerated. *The air must always be cut off from entering the main tube* at *b* when the burner, with the cap *i* attached, is used for a blowpipe-lamp, because if any air mingles with the gas before it is kindled, the flame will be very deficient in reducing power, part of the hydrogen and carbon of the gas being at once oxidized by the oxygen of the air. The cap *i* is easily removed, when the clean, non-luminous, bluish flame is required for ordinary heating purposes, by seizing it with a bit of cloth to protect the fingers from the hot metal. The top of *i* must slant downward somewhat, and the slit in it, through which the gas flows, should be about fifteen millim. long and one millim. wide. It is much better to have this cap fit over the end *c* than inside of it, as the gas is less likely to leak out. The little projection at the right of the slit in the cap *i* may be of service when *i* is used with the high Bunsen burner, presently to be described; but is not at all necessary with the low burners. The top of the cap *i* should, when fitted on the burner, be from ninety-five to at most one hundred millim. above the table. The cock controlling the flow of gas should be within easy reach of the operator, and if the construction of the laboratory table does not admit of this it will be found very convenient to have a control cock on the burner itself, although burners of this style are not usually provided with one.

When the ordinary high form of Bunsen burner, Fig. 3, *B* (a little more than one-fourth the true size), is used, which corresponds in principle to the one already described, having also a movable ring with a hole to cut off or admit air at *m*, the projection on the cap *i* becomes useful in steadying the blowpipe tip, which is rested upon it for producing the oxidizing flame. The high burner is very inconvenient because it prevents the proper steadying of the operator's hands, § 26. In the old style of Bunsen burner the movable ring *m* is often wanting. It is then necessary to provide such a ring or to replace the cap *i* by a long tube *r*, which passes inside of the main, vertical tube *t* and fits over the little, short inner tube through which the gas enters the burner, cutting off the entrance of air at *m* by stopping the hole from the inside. The tubes, *r*, as generally furnished by dealers, have the slit at the top too short and frequently rather too wide.

Brush has recommended (*Determinative Mineralogy*, 1875) a simple blowpipe gas lamp, which is quite inexpensive, although it cannot be used also, like the Bunsen burners, to replace the alcohol lamp. A tube of suitable length and

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about nine millim. in bore is bent at a right angle and made steady by casting a block of lead about the bend, leaving one horizontal projecting end to be connected with the gas supply by a rubber tube, while the other end projects vertically from the lead block. The upper end of this vertical part is flattened and filed slanting, so as to furnish a flat, luminous gas flame, just like that from the cap *i*, Fig. 3.

4. *Gas Blowpipe.* A very handy instrument is shown in Fig. 4. The gas, which may be controlled by a cock at *d*, not shown in the figure, flows through the rubber tube *g* into the chamber *K*, which may be clamped on the tube *a* of the ordinary blowpipe Fig. 1, by a screw *s*, or may be permanently fixed on its own blowpipe tube. The air issuing from the blowpipe tip, mingled with the gas in *K*, passes out through *o*, yielding a long, blue, pointed flame for oxidations, or a less pointed, luminous, mainly yellow flame for reductions, according to the pressure of the blast and the amount of gas admitted.

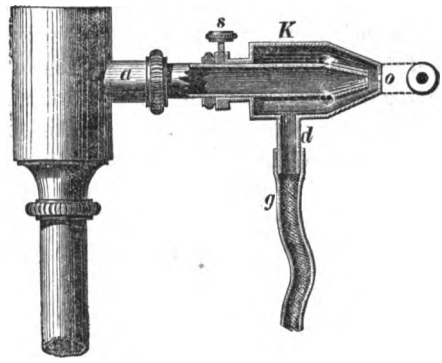

Fig. 4.

5. *Alcohol Lamp.* A small one is nearly indispensable unless gas and Bunsen burners are at hand. The wick should pass somewhat loosely through the socket, and a close fitting cap is needed to prevent evaporation.

III. Supports.

6. *Charcoal.* This must be well-burned, compact but not too hard, of light wood, bass-wood, pine, alder or willow, and must yield only a little grayish or white ash, since a colored ash may lead to mistakes in qualitative work.* It is cut into prisms about ninety millim. long and twenty-five square, so as to show, on two opposite side faces, the edges of the rings of annual growth, which should be

* Foster recommends, in case of need, the use of hard baked clay blocks similar in shape to the coal prisms, but with a cavity in one face near the end, in which may be placed a coal capsule, § 138, *c*, or a similarly shaped bit of charcoal. The face of the clay is then blackened with soot by holding it in the oil flame, after which coats are deposited on it, as on ordinary coal. The soot surface is easily renewed, but the coats formed on it scarcely admit of any extended special treatment. (Plattner, 1878.)

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rather close together, and the substances treated are to be laid on one of these faces. When both faces have been used they are cleaned with a knife or rasp, or the block may be sawn through lengthwise. Great care is necessary in using charcoal a second time, to see that no mistakes arise from the presence of some of the substances already treated. Bits of charcoal can be used, except when coats are to be obtained, being held by some proper support. Natural charcoal is far the best for qualitative work. When it cannot be had the artificial coal, § 138, *a*, may be shaped in suitable moulds, readily devised.

7. Powders which are to be roasted are placed in *very shallow* spoon-shaped depressions on charcoal, scraped out with a knife, or the spatula end of the borer, § 10, *f*. When a substance is to be subjected to the O. F. under any circumstances, unless otherwise directed, a very shallow, saucer-shaped cavity, just deep enough to prevent the substance from rolling off, is also used, in order that the air may have free access and the reducing gases from the glowing coal may have as little effect as possible. On the other hand, when reducing, a deeper cup-shaped cavity, generally about four millim. deep and twice as wide is employed, that the air may be kept away and the substance surrounded by the reducing flames of the lamp and coal.

8. *Glass Tubes.* These are best made of thin glass, free from lead and as infusible as possible.

a. Open Tubes are simply cut from tubing about seven millim. wide, by making a scratch with a sharp file and breaking off the tube in lengths of one hundred and ten millim. It is not usually necessary to bend the tube, as shown in Fig. 5, although this may be done to prevent substances from rolling out. The substance is placed near the lower end of the tube and heated in the alcohol flame, always inclining the tube more or less, according to the current of air desired, and using the blowpipe to increase the heat if necessary. Unless especially directed, the flame must not enter the tube, which is heated from the outside, where the substance lies.

Fig. 5.

b. Closed Tubes, Fig. 6, *B*, are about sixty millim. long and six millim. inside diameter. They are most easily made by cutting the tubing into pieces twice the required length, or a little more, heating these in the middle, drawing out the softened glass and sealing the drawn-out end of each piece by melting it in the flame, remov-

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ing the melted glass with the forceps, so as not to leave the closed end too pointed or too thick. It is not necessary to finish the tube so smoothly as shown in the the figure, but the neater the better.

Matrasses, Fig. 6, *A*, made by blowing into and enlarging the lower end of the closed tubes, while the hot glass is soft. They are sometimes more convenient than closed tubes, but the latter will generally replace them and must always be used when substances are to be heated in presence of as little air as possible.

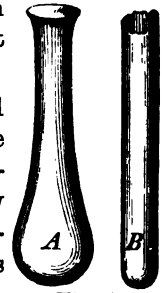

Fig. 6.

c. A Matrass Holder, consisting of two wires, Fig. 7, whose lower ends are fixed in a handle, while the upper ends are bent into two semicircles that can be clamped together by a little ring, moving up and down around the wires, forms a very convenient, but not indispensable, piece of apparatus for holding glass tubes and matrasses.

Fig. 7.

Note.—Sometimes only glass containing oxide of lead can be obtained, and in this case the student must carefully avoid mistakes which might arise from the reduction of this oxide of lead within the glass, forming a dark, lustrous mirror, which resembles closely some sublimates obtained in analysis. The O. F., § 26, must therefore always be used in heating the tubes with the oil lamp.

9. *Platinum Supports.—a. Platinum wire.* This should be about 0.4 millim. thick for general use and may be cut in pieces forty-five millim. long. One end is bent once around the outer end of the conical tube *a*, after removing the platinum tip of the blowpipe, Fig. 1, § 1, the curved end of the wire being brought back to, but not beyond, the straight part, forming a ring about 2.5 millim. in diameter, which is a suitable size for general use with oil lamps. For ordinary work, especially for reductions, with gas, a somewhat smaller loop is better, while quite small loops must be made when candles are employed. Very large loops are to be avoided, except for special purposes.

b. Platinum Wire Holder. This, shown in Fig. 8, *A*, in about one-half the true size, consists of a horn or vulcanite handle, the metal top of which has two cross slits, so that the four divisions thus made can be clamped together by screwing down the hollow cap *a*, Fig 8, *B*. This cap being loosened, by screwing it up a few turns, the wire is passed down through it and between the divisions, where it is securely clamped. This is the Freiberg pattern. The handle of

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the holder receives the spare wires and is closed by the cap *c*, which is screwed on. It is best not to unbend or roughly hammer the wire loops, but to shake off the melted flux from them and then to heat them, looped end upwards, in a test tube with hydrochloric acid, mixed with three parts of water, and finally to rinse them with pure water.

Fig. 8 *A.*

Fig. 8 *B.*

A cork, into which the wires are thrust, may replace the wire holder, but is not to be recommended.

b. Platinum foil should not be too thick, weighing about 110 to 120 milligrammes per square centimetre, cut into pieces forty millim. long and twenty-five wide, and it may be held by forcing its edge into a piece of coal.

c. A thin *platinum spoon*, about 12 to 15 millim. in diameter, with a rather stout handle, will be very convenient for certain fusions. The foil and spoon are best cleaned by fusing in them a little potassium bisulphate, and dissolving out the mass with boiling water. The foil can also frequently be cleansed by simply rubbing it with a piece of charcoal and some water.

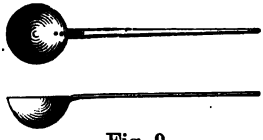
Fig. 9.

Note.—Unless directed to do so, never heat a metal, metallic sulphide, or arsenide in contact with platinum, and be very careful when treating oxides of reducible metals like lead or copper in the fluxes with the reducing flame on platinum wire, since a fusible alloy may be formed with the platinum.

IV. Minor Apparatus.*

(The Freiberg patterns of blowpipe apparatus are the best, with very few exceptions, and they are finished in excellent style by August Lingke & Co., Freiberg, Saxony. S. Hawkridge, Stevens Institute of Technology, Hoboken, New Jersey, also makes reliable apparatus after the Freiberg models.)

10. *a. Forceps with platinum tips*, commonly called *platinum forceps*. Fig. 10 shows the Freiberg pattern, but the simpler French design, Fig. 11, is more durable and generally useful. They are best made of German silver or stiff brass, nickel-plated steel being a very inferior substitute. At the smaller end are narrow platinum tips; the points

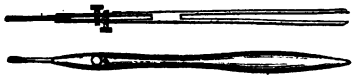
Fig. 10.

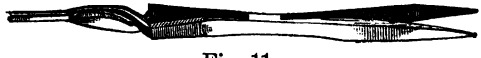
Fig. 11.

* See § 8, *c*, for matrass holder and § 9, *b*, for platinum wire holder.

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of the other end should not be too sharp and narrow. Platinum forceps are necessary in determining minerals, not otherwise.

b. If the Freiberg platinum forceps are used, another pair of German silver or brass *forceps*† will be useful, Fig. 12, for holding small objects under the hammer, since the wider end of the Freiberg platinum forceps is designed rather for flattening out melted glass fluxes than for holding objects under the hammer.

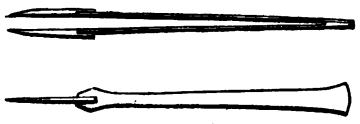

Fig. 12.

c. Steel forceps,† Fig. 13, with points about one and a half millimetres wide, to be used for tending the lamp wick.

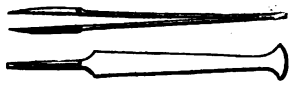

Fig. 13.

d. Scissors,† with narrow and very sharp blades, for trimming the lamp wick.

e. A *knife* with two blades, one large, for general use, and another for testing the hardness of minerals or for any rough usage. One blade may conveniently be magnetized.

f. Steel coal borer with spatula end, or *spatula borer,*† Fig. 14, about one hundred millim. long, with two crossed and slightly curved chisel edges, each six millim. long; the other end a rather stout spatula, nine millim. broad, with a moderately thin, sharp edge. It is a common fault to make this edge too blunt. The chisel end is for boring deep holes; the spatula end for making wider, shallow cavities, or for use as a spatula. If used for quantitative work also, the chisel edges must be of the dimensions given, but this is not so important for qualitative work alone. The spatula borer is necessary for quantitative work and convenient for qualitative.

Fig. 14.

g. A *small steel hammer,*† Fig. 15, having a square face on one end, and a chisel edge on the other, at a right angle with the handle.

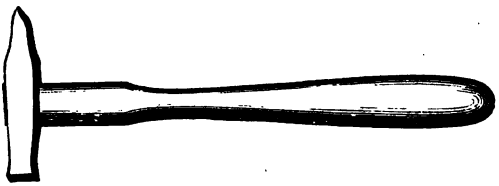

Fig. 15.

h. A small *anvil*† of hard polished steel, Fig. 16, which must be kept clean; see § 11, *b*.

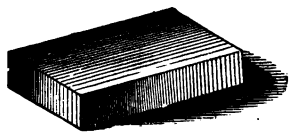

Fig. 16.

i. A *magnifying glass,*† best with two lenses, so as to give a range of power of about five to twenty diameters.

† Apparatus needed also for quantitative assays is marked with a †.

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k. A *charcoal saw*, short and moderately stout.

l. A small strip of blue *cobalt glass*, not "flashed," but blue throughout. It must be of so deep a tint that a strong soda flame, from sodium carbonate on platinum wire in a good Bunsen gas-burner flame, is scarcely visible through it, and when visible has a pure blue color, with no trace of purple or violet. If such glass cannot be had, then two or three thicknesses of the ordinary glass can be employed. As usually sold, one thickness of the glass is rarely dark enough to be of much use in concealing a strong soda flame. The glass may conveniently be mounted in a spectacle frame, or on a little stand, or an arm attached to the blowpipe stand, when the blowpipe is used, but flame tests requiring its use are generally better made with the powdered substance on wire in the Bunsen gas-burner flame. See also § 56.

Fig. 17.

m. Small *porcelain dishes*,† *capsules*,† and *crucibles*,† the latter very thin and light.

n. Watch glasses,† of different sizes.

o. Small *test tubes*,† with a folding, lacquered tin rack, to hold them when in use; a small *wash-bottle*,† two ounces, Fig. 17; the tube *b*, cut in two with a file and reunited with a short rubber tube, so that a stream of water may be sent through it in any direction by blowing into *a*. *Pipettes*† or *dropping tubes*; glass *stirring rods*;† small *glass funnels*,† often conveniently supported in one of the test-tubes.

p. A small *horn spatula* will be useful in certain wet analyses.

q. Files,† triangular and round.

r. Filter-paper,† Swedish, leaving little ash.

s. Small *wide-mouth vials* for holding common, dry reagents; *glass stoppered vials* for acids and wet reagents; *homeopathic vials* for reagents of rarer use. "Specimen tubes" are very suitable for holding reagents, since they do not easily break when packed.

t. An *agate mortar*, with *agate pestle*, is necessary in determining minerals and is exceedingly useful for qualitative work, although the hammer and anvil can generally replace it. It should be about fifty millim. wide for quantitative work, in which it is indispensable; a smaller one will do otherwise. Hard substances must first be broken quite fine on the anvil, before being ground in the mortar. Rubbing with moistened bone-ash will generally cleanse it.

u. Tin flasks with screw caps are useful for holding oil and alco-

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hol. *Rubber stoppers* are best for bottles containing caustic soda or potash and their carbonates, which cause glass stoppers to stick very fast. Glass stoppers that cannot be loosened by gentle hammering with a bit of wood may usually be started by rapidly warming the neck of the bottle in the alcohol flame, withdrawing the bottle before the stopper itself becomes heated, and then turning the latter forcibly.

v. A small *bar magnet,* about eighty-five millim. long, square in section, with a chisel edge at one end.

V. Accessory Apparatus.

11. While nearly all of the above apparatus is needed for a good outfit, a few pieces in addition will be found convenient.

a. Cutting Pliers, Fig. 18, with very stout edges, for nipping off bits of minerals, etc.

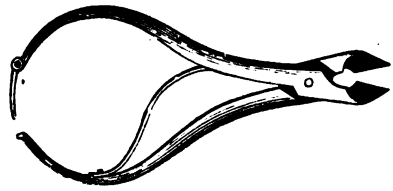

Fig. 18.

b. A *diamond mortar,* of very hard steel, Fig. 19, is useful for crushing hard bodies, when no loss is allowable. The substance is placed within the ring *DE,* fitting into the block *AB,* and is then crushed by hammering on the pestle *F.* Prof. A. R. Leeds has recommended that this mortar be replaced by the anvil, § 10, *h,* in one face of which a hole is bored, into which the pestle fits, without using any ring (*American Chemist,* March, 1877). If the pestle *F* becomes wedged into *DE,* force it out by hammering on a bit of wood placed against the face *C* and never hammer the metal itself. Keep the mortar bright and oiled when not in use, as any rusting makes it work hard and destroys the tight joint necessary between the pieces.

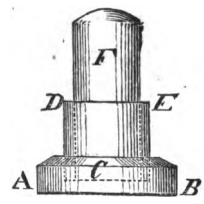

Fig. 19.

c. A *Rasp,* for cleaning and shaping coals.*

(For Quantitative Apparatus see also § 135, *et seq.*)

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* Steel apparatus is less liable to rust if nickel-plated, but the nickel should never be applied to surfaces of hammer heads, anvils, coal borers, spatulas, or forceps used for handling hot fluxes, since the nickel is very apt to scale off when rubbed, and may lead to serious errors. Such pieces of steel apparatus should be highly polished by the maker and *kept clean.* The steel, if only slightly rusted, can be easily polished again with jeweler's rouge.

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VI. Reagents.

Buy all reagents if possible, and let them be chemically pure, unless it is stated that the ordinary articles will answer. Solutions of most chemicals, except acids, are rarely needed in ordinary work, so that many of the reagents may be kept in the solid state until needed, and in cork-stoppered bottles, unless otherwise directed. Reagents marked † are needed also in quantitative assays.

12. Reagents of Frequent Use in Blowpipe Analysis (Fire Tests).

1. *Sodium carbonate, anhydrous.*† (This will hereafter be called *soda*, a name properly applied to caustic soda.) It should be gently ignited in a crucible over the alcohol flame, to low redness, in order to dry it perfectly, and then powdered and kept in a tight bottle.

When used to test for sulphur, or for quantitative copper assays, it must be very free from sulphates. If the test § 103, *c*, or the test mentioned a few lines below, in this paragraph, shows more than the faintest trace of sulphur or sulphates, some pure soda must be obtained as follows :

Put a loose plug of cotton into a funnel, nearly fill the funnel with powdered commercial bicarbonate of soda, cover this tightly with two layers of filter paper, pour on the paper a little distilled water, let it drain through *completely*, add more water and continue this until a little of the filtrate, slightly acidulated with hydrochloric acid, shows no turbidity on addition of a few drops of barium chloride solution. Then dry the residue, ignite and powder it as above. Commercial bicarbonate of soda can often be found pure enough for all purposes except testing for sulphur, and it need only be gently ignited and powdered. If the bicarbonate cannot be obtained, the ordinary carbonate can be purified by repeated recrystallization from hot solution, but the process is more tedious.

2. *Borax*† (*sodium tetraborate* with water of crystallization*). Commercial borax is good if it gives a perfectly transparent, colorless bead, both hot and cold, in O. F. and R. F. (For ascertaining the presence of a little iron oxide as an impurity, see *note* § 74, *a*.) If it does not seem good it must be recrystallized.

Since ordinary borax swells very much in fusing, it is an excellent plan to melt some of the borax to a clear glass in a platinum crucible, pouring it out on a clean, bright iron plate to cool, and then crushing it into coarse fragments in the steel mortar, § 11, *b*, or between paper, sifting out the fine powder and keeping both in tight bottles (*granulated* and *powdered borax glass* †). The *powdered* glass is used whenever borax glass is *mixed* with substances in assays.

* Borax loses its water of crystallization when fused, and the resulting glassy, acid salt readily dissolves metallic oxides.

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Calcined borax, or borax gently heated until it ceases to swell and has lost its water of crystallization, after which it is powdered, is better than raw borax, but not so good as the glass. If the borax is to be used in connection with the wet analysis the recrystallized salt is used, being first fused as above, and its solution in water must yield no precipitate with solution of sodium carbonate.

3. *Salt of Phosphorus* † (*microcosmic salt ; hydrogen-ammonium-sodium phosphate*). The letters S.Ph. will be used to indicate this salt. When S.Ph. is fused, water and ammonia escape, leaving sodium metaphosphate, which readily dissolves most metallic oxides.

S.Ph. must give a perfectly transparent, colorless bead when fused on platinum wire, both when hot and cold, in O. F. and R. F., and must show no reaction for chlorine, § 104, or if it does, the chloride of sodium which occasions it must be separated by recrystallization. It can be made by heating twenty-five parts of crystallized sodium phosphate, four parts of ammonium chloride, and eight parts (all by weight), of water together until dissolved, then filtering the hot solution, cooling it until the S.Ph. crystallizes out, draining off all the mother liquor from the crystals through a filter, drying the salt on filter paper and preserving it in a tight bottle.

4. *Potassium nitrate* or *nitre*. The purer commercial article will answer. It is often used, but in small quantity.

5. *Cobalt nitrate*. The crystals are dissolved in ten parts by weight of water, and kept in a small bottle with a cork, Fig. 20, through which passes a narrow glass tube open at both ends. If the tube is closed above with the finger and withdrawn, together with the cork, a part of the solution will remain in the tube, escaping by drops when the finger is partly removed.

Fig. 20.

The crystals should be carried in a small glass-stoppered bottle on a trip and a minute fragment dissolved in water when wanted.

Cobalt nitrate is decomposed by ignition, and the resulting cobalt oxide combines with certain oxides, producing characteristically colored compounds.

6. *Potassium bisulphate* † (*hydrogen potassium sulphate*).

Heat gently in a porcelain crucible one part by weight of potassium sulphate with one-half part of strong sulphuric acid, and gradually increase the heat, stopping as soon as the mixture ceases to boil violently and subsides to a fused, liquid mass, clear as water. When it is cold, the white, opaque mass is shaken from the crucible, coarsely crushed and kept in a glass-stoppered bottle. The commercial bisulphate is pure enough for most work.

7. *Boric acid*† (*boracic*) fused to a glass and kept in a tight bottle.

8. *Copper oxide*, commercial, in fine powder. It can be made by

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dissolving copper in nitric acid, evaporating to dryness, and igniting the salt in a porcelain crucible until it is black.

9. *Test lead.*† It must be especially free from gold, silver, and copper, and can be prepared by placing a piece of zinc in a strong, clear solution of lead acetate, detaching the spongy precipitate of metallic lead from the zinc every few hours, and finally washing it repeatedly by decantation with hot water, drying it thoroughly between filter paper, and finally over the lamp in a porcelain dish at a very gentle heat. If required for qualitative assays some of the lead must be fused and hammered into sheets. For quantitative work it may be used in the original powder, after this has been rubbed in a mortar and sifted ; see also § 148, *a.*

10. *Silver.*† A moderately thick bit of foil (or a coin) to be used for the sulphur test, § 103, *c;* also some pure silver foil, rather thick, to be cut into small bits as needed. In this shape it is almost exclusively used for quantitative assays. It is best prepared by reducing silver chloride by fusion with sodium carbonate, § 148, *d.*

11. *Tin,* best in thick ribbon. Thin foil, if used, must be tightly rolled and hammered. A small bit of the tin fused for a moment on coal in a reducing flame, in contact with an already made bead of S. Ph., § 46, *c,* must impart to it no dark tinge, due to antimony.

Tin has a strong affinity for oxygen at high temperatures, and readily abstracts it from reducible oxides when fused in R. F. beside glass fluxes containing such oxides. On this account it facilitates the reduction of sesquioxide of iron to protoxide, of protoxide of copper (cupric oxide) to the suboxide (cuprous oxide) ; of bismuth oxide to metallic bismuth, etc., and is therefore a very valuable blowpipe reagent. It also serves to effect reductions in the wet way, through liberation of hydrogen, when heated in certain acid solutions.

12. *Gold.* Obtained in a pure state by cupellation and parting with silver and nitric acid ; see the quantitative gold assay.

13. *Potassium iodide* and finely divided *sulphur* (*flowers of sulphur* best). Equal volumes of each well mixed.

14. *Bone-ash.*† Bones are burned, the perfectly white parts crushed, ground, and sifted through a fine hair sieve. A portion of this is kept for quantitative silver tests and a portion is elutriated, § 21. The very fine elutriated or *washed bone-ash* is kept for qualitative and quantitative work, after being dried and heated for some time to low redness, while the coarser part is again ground and elutriated. Bone-ash should be kept in tight bottles.

13. Reagents Less Frequently Used in the Solid State.

1. *Potassium oxalate, neutral;* it may be useful as a reducing agent, or in quantitative nickel and cobalt assays.

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2. *Potassium cyanide;* it must be kept in a tight bottle. It is sometimes the best reducing agent, but will very rarely be needed.

3. *Potassium pyroantimonate.* Mix one part of finely powdered antimony with six of nitrate of potash, and throw it, a little at a time, into a red-hot clay crucible. As soon as it is well fused pour it upon the anvil, powder it, wash the powder several times with cold water, boil the residue with water, evaporate the filtered solution to dryness and strongly heat the dry mass. Powder it rapidly and keep it in a glass-stoppered bottle, if it is likely to be often wanted. It is a reagent very rarely employed, being used only for detecting carbon. If its solution in water, when heated, gives off any bubbles of gas on addition of nitric acid, the salt cannot be used to detect carbon.

4. *Potassa*† (*potassium hydroxide* or *caustic potash*). It should be chemically pure, and is best kept in a wide-mouthed vial with a rubber stopper.

5. *Potassium carbonate,* commercial; sometimes useful in testing for iodine.

6. *Silica*† (*powdered quartz*). Sometimes useful in testing for sulphates; also in the quantitative separation of tin from copper, and in testing for phosphates.

7. *Nickel oxalate,* or the *oxide* free from cobalt; rarely used in testing for potassium.

8. *Bismuth oxide;* used in some tests for iodine.

9. *Sulphur*† (*flowers*). Used in some tests for iodine and occasionally in quantitative copper assays.

10. *Calcium carbonate* (*calcite*) free from alkalies. It is useful in decomposing silicates containing the alkalies.

11. *Ammonium chloride,*† commercial resublimed. It is used in connection with calcium carbonate in testing for the alkalies; also in separating platinum from gold.

12. *Fluorite* (*fluor spar*) is sometimes used in testing for lithium; also to make Turner's flux.

13. *Gypsum* (*calcium sulphate*) may be used in testing for lithium.

14. *Iron wire,*† as stout as a medium knitting needle. It is sometimes useful in testing for antimony and is needed for the quantitative lead assay.

15. *Ammonium carbonate.*† It is useful in certain tests for uranium and in roasting ores; also in wet analyses.

16. *Mercuric chloride* (*corrosive sublimate*). Occasionally used in testing for iodine.

17. *Starch powder.*† It is used in the wet analysis for iodine and in certain quantitative assays to make Plattner's flux.

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18. *Turner's flux.* Four and one-half parts of powdered potassium bisulphate mixed with one part of finely powdered fluorite (fluor spar). It is used in testing for boric acid and must not color the flame green when treated alone on wire, § 113, *a.*

19. *Sodium hyposulphite* (*thiosulphate*). Used in some tests for cyanogen.

20. *Magnesium,* in thin wire or ribbon. It is used in testing for phosphoric acid.

21. *Litharge*†; used in certain tests for nitrates.

22. *Zinc,* commercial. It serves in certain reduction tests of solutions for tungsten, columbium, etc.

14. Liquid Reagents.

1. *Sulphuric acid.*† The concentrated acid is often used in testing for phosphoric and boric acids, and is a useful reagent in many wet analyses.

2. *Nitric acid.*† When diluted with water it should yield no turbidity with silver nitrate solution.

3. *Hydrochloric acid.*† When diluted with water it should yield no turbidity with barium chloride solution.

All of the above acids are understood to be diluted with four parts of water, when "dilute" acids are mentioned; otherwise the concentrated acid is indicated.

4. *Aqua regia.* Hydrochloric acid containing about one-third part of nitric acid. The effect of aqua regia is obtained by adding to the hydrochloric acid, when used, successive portions of nitric acid, until the desired effect is produced, and it is not usually best to prepare any large supply.

5. *Acetic acid.* Of limited use in certain wet analyses and in removing bone-ash and litharge from silver buttons.

6. *Hydrosulphuric acid* (*sulphuretted hydrogen*). It is used only in wet analyses and may be employed either as a solution of the hydrosulphuric acid gas in water, or in the form of gas.

The gas is generated by pouring dilute sulphuric or hydrochloric acid on fragments of sulphide of iron covered with a little water in a bottle or test-tube. The bottle is provided with a cork through which passes a rather wide bent tube, the end in the bottle being loosely plugged with wet cotton to purify the gas. The gas issuing from the other end may be passed at once into the liquid to be treated, or may be passed into water, until this is saturated with the gas, thus furnishing hydrosulphuric acid solution. To ascertain when any liquid is saturated with the gas, remove the liquid from the gas-generating apparatus, blow away any gas floating in the vessel and stir the liquid. If it is saturated it will still smell of

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the gas, or will darken filter paper slightly moistened with solution of lead acetate and held in the vessel, *just above* the surface of the liquid, not *in* the liquid.

7. *Ammonium sulphide* is made by saturating moderately dilute ammonia water with hydrosulphuric acid. It is used only in wet analyses.

8. *Ammonia water* or *ammonia.*† It is used in many wet analyses, especially in determinative mineralogy.

9. *Ammonium molybdate.* Very useful in testing for phosphoric acid. The solution is prepared by dissolving, with the aid of heat, one part of molybdic acid in eight of strong ammonia and pouring the cooled solution into ten parts of strong nitric acid, previously mixed with as much water. The solution should be allowed to stand until it becomes clear.

10. *Alcohol, absolute.* It is used in wet analyses to separate barium, strontium and calcium; also in testing for potassium and in separating platinum from gold.

11. *Chloroform* is sometimes used in testing for iodine and bromine in the wet way.

12. *Distilled water*† is generally useful. Pure rain water is usually good enough for wet analyses.

13. *Glycerine* may sometimes be useful in detecting boric acid.

14. *Platinic chloride.* Platinum scraps are purified, if need be, by boiling with hydrochloric acid and washing with water, after which they are dissolved in hot hydrochloric acid with addition of a few drops of nitric acid from time to time. The solution is evaporated on the water bath to expel free acid and the dry residue dissolved in about ten times its weight of water and the filtered solution kept for use in detecting potassium, and possibly ammonium by the wet way.

15. Solid Reagents Only Used for Wet Analysis.*

1. *Tartaric acid,* used especially in separating tin and antimony.

2. *Citric acid* furnishes, according to Prof. H. C. Bolton (*Annals New York Acad. Sci., Vol.* I, 1877–78), a convenient means of testing for carbonates and decomposing many minerals.

3. *Barium chloride;* used in testing for sulphates.

4. *Barium nitrate* replaces barium chloride when silver, lead, or mercurous salts are in solution.

5. *Copper sulphate;* it may serve in certain tests for chlorine and bromine.

* They may be kept in the solid state and dissolved in water when needed.

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6. *Ferrous sulphate*† (*protosulphate of iron*) is used in testing for nitric acid in the wet way and sometimes in determining gold in the wet way.

7. *Lead acetate* is used especially in testing for chromium and phosphoric acid.

8. *Ammonium oxalate;* used in testing for calcium.

9. *Magnesium sulphate;* used in testing for phosphoric and arsenic acids.

10. *Potassium permanganate;* see § 56.

11. *Oxalic acid;* used in detecting cerium.

12. *Potassium bichromate* serves for detecting chlorine in presence of bromine; also sometimes in testing for barium.

13. *Potassium sulphate;* dissolved in two hundred parts of water it serves to separate strontium from calcium.

14. *Sodium phosphate* may be used in testing for magnesium.

15. *Silver nitrate;* as a test for soluble chlorides.

16. Test Papers.

1. *Blue litmus paper.*† Heat one part of commercial litmus with six of water gently for some hours, adding water at times to maintain the original volume. Filter, divide into equal parts, stir one part with a glass rod dipped in very dilute sulphuric acid, until the liquid is just reddened, and then mix the two parts. Dip filter paper into the liquid and hang it up to dry in pure air. The paper should have a bluish color, not too deep, and should turn red when dipped in water containing very small quantities of a free acid.

2. *Red litmus paper.*† Prepare litmus solution as at first, and, without dividing it, stir it with a rod dipped in dilute sulphuric acid until it is just distinctly red, but no more. Prepare paper with this as before. It should be pale red and turn blue with very dilute alkaline solutions.

3. *Turmeric paper.* Digest, with the aid of heat, one part of powdered turmeric and six of moderately dilute alcohol; filter, and dip filter paper into the filtrate. When drained and dried the paper should have a bright yellow color, which is changed to a reddish-brown by alkalies; to a peculiar red by boric acid; to orange-yellow by zirconium solutions.

4. *Brazil-wood paper.* Boil the shavings with water, filter, and prepare paper with the filtrate as above. It is used in testing for fluorine.

Note.—Test papers of different kinds should not be placed in contact with one another.

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17. All of the preceding reagents may occasionally be needed and will be at hand in a well equipped laboratory, but for the general qualitative work laid down in the introduction, and for determinative mineralogy, the following will suffice: § 12, the entire list; § 13, 4, 6, 10, 11, 12, 15, 18, 20, 22; § 14, 1, 2, 3, 6, 7, 8, 9, 12, 14; § 15, 4, 7, 8, 11, 14, 15; § 16, the entire list.

The most necessary reagents, such as would be carried on a trip, are the following, of which the quantities indicated may be provided. Even of these, the ones marked ‡ can be generally omitted:

One ounce each of § 12, 1, 2 (the granulated glass); 8, 9 (granulated or finely divided); 14 (washed). § 14, 3. One-half ounce of § 12, 7. Of the following a small homeopathic vial full: § 12, 4, 5 (even less); 6, 8 (even less); 10 (one piece), 11, 13; § 13, 4‡, 10‡, 11‡, 12, 20‡; § 15, 3‡; § 16, 1, 2 (or 3). It will be convenient to have § 14, 1, unless § 13, 20, is taken, to test for phosphates. § 14, 2 and 8, are useful, but not indispensable, unless 2 is needed in testing for gold.

All liquid reagents should be packed in a box by themselves, and the glass stoppers tied down firmly. Solid reagents can be carried in a stick of wood, provided with the necessary holes, having tight cork stoppers; but caustic potassa acts rather easily on cork, as does cobalt nitrate. A neater way is to keep the reagents in homeopathic vials, which can be fitted into holes in the wood and kept in place by a sliding cover.

VII. General Operations in Wet Analysis.

18. *a. Solution.* This proceeds most rapidly when the substance under treatment is finely divided, in powder or filings, and, unless forbidden, it is generally best to heat the solvent. Test tubes are very convenient for making solutions, as they can be very readily heated, the only precaution being to shake the tube gently in the flame, which must not strike the tube above the liquid. Sometimes a substance is only partially soluble; to determine then whether anything has dissolved, filter a little of the solution and evaporate it slowly to dryness on clean platinum foil, to see if any residue remains. A solution may be colored or colorless, but if complete will always be perfectly transparent. An *aqueous solution* means a solution in water. A solution is neutral when it has no reaction on either red or blue litmus paper.

b. Evaporation. This should be conducted in a wide vessel and at the highest heat below boiling, unless otherwise directed. Sometimes a *water-bath* is useful, since the heat cannot then rise too high. A beaker may be used as a water-bath, the evaporating vessel being placed on the beaker so as to be heated by the steam from the boiling water in the beaker. When a solution has been sufficiently concentrated by evaporation, any crystallizable substances in it will separate in crystals, more or less distinct, as the solution cools, and when substances of different solubility are present one of

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them may be generally thus separated from the other by crystallization, which can be repeated until the desired substance is purified from the more soluble substances, which will remain in the original solution, then called the *mother liquor*.

19. *Precipitation.* On adding to a clear solution a proper reagent, the substance in solution will form an insoluble compound which goes down as a *precipitate*, rendering the solution cloudy or turbid. Some precipitates form at once, others slowly; some are granular, others pulverulent, crystalline, curdy, flocculent or gelatinous (like jelly). The concentration of the solution often affects the form and rapidity of appearance of the precipitate. Unless otherwise directed, solutions should be moderately concentrated if precipitates are to be obtained, and it must be borne in mind that precipitates form in concentrated solutions when they may not in dilute solutions. Too concentrated solutions may, however, lead to mistakes. Thus concentrated hydrochloric acid will produce a precipitate in the reagent solution of barium chloride, simply because both are too concentrated. Unless forbidden, it is better to precipitate from hot solutions and keep nearly boiling for a while afterwards, to facilitate the separation and filtration of the precipitate.

The reagent and solution must be well mixed by stirring or shaking, and after the precipitate has settled a drop of the reagent must be carefully run down the inside of the vessel, to see if it produces a further precipitation. If the precipitate does not settle readily a drop of the filtrate must be tested with the reagent.

20. *Filtration.* A circle of filter paper is cut, whose radius is somewhat less than the length of the funnel side, and is first folded into a semi-circle and then into a quadrant. This is opened so that it forms an inverted cone, with one thickness of paper on one side, and three thicknesses on the other, and is then placed inside of the dry funnel, with the apex of the paper cone in the apex of the funnel, the whole fitting snugly to the glass. The paper is then moistened thoroughly with water from the wash-bottle, and this is allowed to drain out. The wetting must never be omitted unless directed, as precipitates easily clog the pores of dry filters. The funnel is now placed in its support and the solution decanted into the filter, the stream being guided by a stirring rod which has already been dipped in the solution to be filtered. It is highly desirable to run on the clear solution first, and finally the precipitate. The filtered solution is called the *filtrate*.

21. *Decantation, washing precipitates,* and *elutriation. Decantation* means pouring off the clear solution from the insoluble residue.

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Precipitates which settle rapidly should be kept in the original vessel, a little water added, hot if admissible; the precipitate stirred thoroughly with it, again allowed to settle, the wash water decanted, (into a filter if necessary), and this "washing by decantation" repeated several times, when the precipitate is itself finally brought with the last wash-water upon the filter. To remove any of the precipitate which adheres to the vessel, hold the latter partly inverted over the edge of the funnel, and direct a stream of water from the wash-bottle upward into the vessel. Sometimes the precipitate must be detached by rubbing with a little bit of rubber tubing slipped over the end of a stirring rod, or with a small end of a feather. If necessary, the precipitate must be washed after collecting it on the filter. This is done by directing a stream of water from the wash-bottle upon the precipitate, so as to wash all down into the apex of the filter, filling the filter only about half full of water, and allowing all of this to run through before adding more wash-water. A precipitate is clean when a few drops of the wash-water running through the filter and evaporated on platinum foil leaves no residue. Eight or ten washings carefully made with hot water will generally suffice. The wash-water is usually thrown away in qualitative analyses.

Elutriation consists in stirring a powder with water, allowing the coarser particles to settle a few moments, and pouring off the finer particles suspended in the water into a second vessel, where they may settle completely.

22. *Drying precipitates.* After draining off the water, the funnel and its contents are placed over the lamp and gently dried, or the filter paper may be carefully removed, opened, and dried in a porcelain dish, taking great care not to char the paper, from which the dry precipitate is removed, sometimes by scraping, sometimes by rubbing the paper together. Frequently in qualitative work a precipitate can best be removed by opening the moist filter on two or three layers of filter paper, and scraping off the precipitate with a spatula. If the precipitate is to be re-dissolved, this may often be done by pouring the solvent upon it at once in the funnel, collecting the solution, and again pouring it through the filter, until the precipitate is all dissolved. It is often well to make a hole in the filter with the stirring rod and simply wash the precipitate down into a vessel, decanting the water afterward if necessary.

23. *Ignition.* Precipitates are separated from dried filters, placed in a crucible, the filter paper rolled up with any adhering precipitate inside of it, the roll ignited thoroughly on the cover of the crucible, over the alcohol lamp, and the ash, perfectly free from carbon, placed

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with the precipitate in the crucible. This is now covered, set on the wire triangle, and gently heated over the spirit lamp, the cover is then partly removed and the crucible and its contents brought to a bright red heat. Most precipitates will be sufficiently ignited in fifteen minutes. The carbon is all consumed when the filter ash shows no traces of black; but if the precipitate is itself black after ignition, the operator must depend on his experience and judgment. The flame of the alcohol lamp must not enter the crucible. Sometimes a blast from the blowpipe will be necessary to ignite the precipitate thoroughly, but generally the alcohol flame will suffice for the small quantities treated in our analyses.

Ignition is never performed except in thin capsules, crucibles, on platinum foil, or on coal, since the glass or thicker porcelain vessels are liable to crack, and no vessel containing any moist substance should be heated strongly.

24. *Cleansing and preserving apparatus.* All vessels should be cleaned before they become dry, as the dirt may generally then be easily rubbed off with a bit of sponge tied to a slender whalebone. If not easily rubbed off, try some proper solvent; hydrochloric acid is generally sufficient. All metallic apparatus must be washed and wiped dry after use, and steel should be oiled when laid away for any length of time. Very fine emery paper is good for removing thicker coatings of rust; jewelers' rouge for slight deposits. Only the most scrupulous cleanliness will secure the operator from mistakes in analysis.

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CHAPTER II.

GENERAL OPERATIONS AND EXAMINATIONS IN QUALITATIVE BLOWPIPE ANALYSIS.

I. Management of the Flame.

A. The Blast.

25. The student can only succeed with the simplest substances, unless he learns to sustain a powerful and uninterrupted blast for several minutes. Properly managed, no fatigue will be felt at the end of a long operation. Let the student place the blowpipe with its platinum jet and trumpet mouthpiece attached, firmly against his closed lips, distend his cheeks *fully* with air from the lungs, and while they are thus distended, fill and empty his lungs several times through the nose, paying no further attention to the blowpipe, but keeping the cheeks fully distended all the time. This is easily done. Let him repeat the operation, this time holding the platinum jet against the flame of the alcohol lamp and allowing air to pass from the lips through the blowpipe. He will find that a blast from the blowpipe is constantly passing through the flame, and the supply of air in the cheeks will last during one or two inhalations, being then renewed with only the slightest interruption and without letting the cheeks collapse, by allowing some air to enter the mouth from the inflated lungs, the greater portion of their contents being, as before, exhaled through the nose. The whole secret of producing the continuous blast without fatigue lies in the fact that the muscles of the fully distended cheeks mainly produce the blast, the lungs serving only to force air from time to time into the distended cheeks. If the student tries the apparently simpler method of fully inflating his lungs and keeping up the blast from them alone, as long as that one supply of air lasts, he will soon be out of breath, and the flame must inevitably cease entirely during each inhalation. Physical malformation alone can permanently interfere with the necessary use of the nostrils, and in such cases either the simpler tests only can be made, or automatic apparatus must be used, the best of which is bad for qualitative work.

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B. The Oxidizing Flame (O. F.).

26. Place the oil lamp, § 2, Fig. 2, on the table, its reservoir clamped so that the wick is about four inches from the table, trim the already well charred wick clean and parallel with the slanting, upper edge of the socket, the highest part to the right, the reservoir of the lamp at a right angle with the edge of the table. Hold the blowpipe firmly in the right hand, with the fore and middle fingers above the tube, a little below its middle point, and the thumb and two other fingers supporting it beneath ; with the right fore-arm resting against the edge of the table, not with the elbow upon the table. This position best secures the steadiness essential to successful work. Direct the alcohol flame upon the oil wick with the blowpipe, and when lighted raise the wick so high that the flame just does not smoke ; a small flame is too feeble, a smoking one dirty. As it is very necessary to hold bodies steady in the flame the oil lamp should be so high that the left hand, when holding the wire holder or other instrument, may rest with its little finger on the table.

To produce the *Oxidizing Flame* introduce the blowpipe jet about half way across the wick, nearly, but never quite, touching it, and blow a rather strong blast downward, and in every way exactly parallel with the wick. This should produce a long, blue, sharp-pointed flame, perfectly steady, free from yellow streaks and unaccompanied by any hissing noise ; defects caused by a ragged wick, ill-shaped or dirty orifice of the jet, contact of the jet with the wick, or wrong direction of the blast. A perfect O. F. consists of a blue cone, extending from *b* to *a*, Fig. 21 (in a sufficiently powerful flame it will be about 20 millim. long), generally surrounded near the blowpipe by a small luminous, yellow flame, which must not extend far down the cone, and a faint bluish-red or reddish-yellow, nearly invisible flame, extending from *a* to the very tip, *c*. The blue flame is that of burning carbon monoxide and hydrocarbons, and the student must remember that it possesses strong *reducing powers*, § 27, although it is not well suited for reductions, because it is not large enough to protect bodies from the air. This blue cone, of great use in getting colored flames, § 42, will be called the *inner flame*.

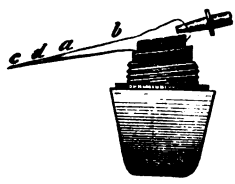

Fig. 21.

Just beyond the apex *a*, of the blue inner cone, at *d*, the flame is hottest, and this part is in general used in determining the fusibility of substances.

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The *strongest oxidizing action*, which results from the exposure of the heated body to the free action of the air, is obtained just beyond the point *c*, ten to fifteen millim. from the tip of the inner blue cone of flame, *a b*, since bodies can still be kept very hot here, while no reducing gases reach them. It is, in general, a good rule when oxidizing substances in the fluxes to keep them as far from the blue inner flame as possible provided the flux remains red-hot and perfectly fused.

When gas is used with one of the gas burners, § 3, the oxidizing flame is obtained in a similar way. The gas flame should not be too large, and it will be found that the most perfect oxidation is obtained at a point rather more distant from the tip of the inner blue cone of flame than is the case with oil.

C. The Reducing Flame (R. F.).

27. Withdraw the blowpipe, until its jet scarcely projects over the wick, and raise it a little higher above the wick, blowing less strongly, so as to produce a thicker, less pointed, luminous, yellow flame, Fig. 22, in which the blue inner cone, from *b* to *d*, is much shorter than in the O. F., being often scarcely perceptible. The flame from *d* to *a*, or even the entire distance from the left-hand side of the wick to *a*, about 30 to 35 millim. in a good flame, is nothing more than the ordinary oil flame, deflected from its upward course by the blast, and forming a nearly solid, yellow, somewhat hotter flame. Occasionally a very faint, mantling flame is visible also around its apex at *c*. A substance held in the solid cone of luminous, yellow flame, almost midway between *a* and *d*, but nearer *a*, is strongly heated in contact with carbon monoxide, gaseous hydrocarbons and probably incandescent particles of carbon, all of which have a strong affinity for oxygen, abstracting it, if possible, from the substance, which is thus *reduced*, sometimes to metal, sometimes only to a lower stage of oxidation. (The *reduction* of a compound signifies the abstraction of oxygen, chlorine, sulphur, or some other *acid* or *acidific* element from the *metallic* or *basic* element; the reduction being total when oxide of copper is reduced to metallic copper, partial when binoxide of manganese is reduced to protoxide. The reduction may be effected by some reagent, as when chloride of mercury is reduced to metallic mercury by being heated with carbonate of soda, in which case chloride of sodium is also formed.)

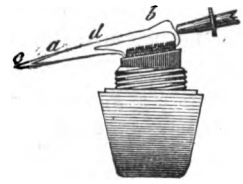

Fig. 22.

In order to exclude outside air, which would have an oxidizing

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action, take care to keep the substance *completely enveloped* in the luminous flame, and sustain throughout the operation an *uninterrupted* blast. This must not be too feeble, so as to yield a very sooty flame, nor too strong, since then the air forced through the blowpipe may oxidize the substance. Keep turning the substance slowly, so as to present both sides of it to the lamp, and do not allow any considerable deposit of soot to form on it, for this retards the operation and must be burned off by blowing a little harder. Do not bring the substance too near the blowpipe, into the dark cone of blue flame, for here it may be partly re-oxidized. Keep the lamp wick pressed together, and when undertaking a very difficult reduction on platinum wire, raise the wick so that the flame may smoke slightly, thus ensuring a sufficient body of flame.

In making reductions on coal the student will find it generally advantageous to revolve the body of the lamp, when its construction admits of this motion, as it should, so that the flame may be pointed more downward, and to blow a somewhat stronger blast, since the coal will thus be made to glow more brightly and the gases generated from it will greatly aid in the reduction. A somewhat higher temperature is generally useful in reductions on coal.

When gas is used with one of the burners, § 3, the reducing flame is obtained in a similar way to that just described for the oil lamp. The gas is turned on so as to give a large flame (even seventy millim. high, when any great heat is required), and the blowpipe jet being held just outside of the flame, and at some distance above the top of the burner, four to seven millim. according to the style of burner and size of the flame used, a moderately strong blast is directed so as to give a yellow, reducing flame, just as described with the oil lamp. This flame will serve for reductions on wire with moderately difficult tests, but is much inferior to the oil R. F., and it will often be advantageous to reduce the substance on coal instead of on wire, securing thereby the aid of the reducing effect of the glowing coal; a stronger blast may then also be used than with wire.

D. Testing the Oxidizing and Reducing Flames.

28. The student must carefully test his power to manage the flames.

a. Heating power of the O. F.—Bend a bit of platinum wire, 0.1 millim. thick, at a right angle, hold the shorter end steadily within the hottest part of the flame and parallel with it, and fuse the extreme end to a minute globule.

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b. Purity of the O. F.—Make a borax bead and dissolve in it, § 44, finely powdered molybdenum trioxide (molybdic acid) [1], in sufficient quantity to make the bead decidedly yellow while hot. The O. F. must be used, and care will be necessary, even for this purpose, to keep the bead far enough from the point of the inner blue cone *a*, Fig. 21, § 26. When everything is dissolved and the bead is quite transparent, except perhaps a few exceedingly small, dark specks of the undissolved oxide, treat it in R. F. It should become almost black, when cold. If it does not, repeat the treatment in O. F., after adding a little more of the oxide, and again reduce it. When the dark bead has been thus obtained, try to render it transparent again with the O. F. The sooner this can be done, the better is the O. F. used. The bead will be nearly or quite colorless when cold, after proper oxidation, and perfectly transparent. The student will find that the least contact of even the blue flame will darken such a bead more or less, and therefore the bead should be held as far from the flame as possible, but still kept in perfect fusion, while the attempt must not be abandoned without success. The brown color results from the reduction of the molybdic oxide (MoO_3) to a binoxide (MoO_2) and the O. F. again raises it to MoO_3.

c. Purity of the R. F.—Dissolve in borax on a clean platinum wire in O. F., enough *finely* powdered binoxide of manganese [2] to render the bead decidedly violet-red when cold. The bead must show no black specks, resulting from too coarse powder or too short treatment with the O. F. Keep this bead in the R. F. (which must be rather large) well surrounded by the yellow flame, turning it slowly so as to present different sides of it toward the lamp, but not letting the air reach it, until it finally becomes colorless on cooling, owing to the reduction of the manganese sesquioxide (Mn_2O_3) to the protoxide (MnO), which has no perceptible coloring effect. So long as the bead contains any of the highly coloring sesquioxide it will, while red-hot, also appear opaque, but when only the protoxide remains, the bead, still kept in the flame, becomes transparent.

Having effected the reduction of a moderately charged bead, the student should try again, this time adding so much of the binoxide that the bead, in O. F., is very dark red, the lamp flame being only faintly seen through it. This time it will be necessary, as soon as the red-hot bead in R. F. becomes transparent, showing the complete reduction to protoxide, to stop the blast and quickly flatten the bead somewhat between the hammer or the end of the blowpipe and the anvil, to cool it before the air can reoxidize the protoxide. The cold bead should now be clear and colorless. The student can scarcely

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expect to succeed as well in this more difficult test with gas as with oil, but he should be able to reduce a rather highly charged bead even with gas.

Note.—If the borax becomes volatilized, through too much time being consumed in the preceding tests with O. F., make new beads, that the film of borax may not be too thin to afford a fair test.

d. Heating power of the R. F.—Dissolve in borax on platinum wire in O. F. enough oxide of copper [3], in successive portions, to make the bead finally opaque. Detach the bead from the wire, § 44, 1, and treat it on charcoal, in a moderately deep cavity, with R. F., until the reduced metallic copper collects into a button, leaving the borax colorless. It will require a hot and yet pure R. F., produced by a rather strong blast, to melt the copper so that it will collect to a button, which can be picked out from the borax, either while the latter is still soft or after it has been crushed between paper. If gas is used the borax bead must not be too large or it will be found very difficult to reduce the last traces of copper suboxide, which would otherwise color the cold bead reddish.

Do not try any other tests until these have been satisfactorily accomplished, as only failure will result in many cases. Skill in the management of the O. F. and R. F. is essential to successful analytical work.

II. General Examinations.

The student must render himself thoroughly familiar with these, using the trial substances named in each paragraph, the numbers in square brackets referring to the list in the Introduction.

A. Examination in the Closed Tube.

29. In many cases characteristic results are obtained here; in others only such as show the presence or absence of various bodies which give more or less similar results.

As a general rule, it is best to use small quantities of the substance and in moderately fine grains, a fragment about half the size of a hemp-seed, broken into a few smaller grains, being usually sufficient, although decrepitation is best observed when the substance is not quite so finely divided, while the evolution of gases is usually favored by powdering the substance, and this may be necessary in case of bodies yielding only a little water and that at a high temperature.

The tube, § 8, *b*, is examined with the magnifying glass, to avoid any misleading phenomena, such as apparently crystalline fragments,

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etc., and cleansed if necessary with a roll of soft paper. Place the substance in the bottom of the tube, or if in powder, crease a narrow strip of paper lengthwise, put the powder on one end, push this end down to the closed end of the tube, tip the latter and withdraw the paper, leaving the powder at the bottom of the tube, the inside of which must be cleansed again if need be. Clamp the tube in the matrass holder, § 8, *c*, and heat the lower end of it, at first very gently, in the alcohol flame, and finally to redness, using a blast from the blowpipe if necessary. The tube should be slightly inclined and must not be heated in the upper part, where substances may condense when cold. Consult note, § 8. Moistened test papers, in narrow slips, are introduced into the tube whenever the action of the vapors, etc., on them is to be ascertained. Before heating an unknown compound in the tube it is well to heat a little of it on coal B.B., to see whether it is explosive, although this is not necessary with minerals.

The successive phenomena are carefully noted, as the temperature rises, or the time of treatment is extended.

30. *a. The substance decrepitates,* or flies to pieces, as do many minerals and salts; *glows* as if on fire for a moment, *gadolinite* and a few other minerals; *phosphoresces* at a moderate heat, some specimens of fluorite [4] and apatite do this to a marked extent; it is frequently observed more or less strongly in the case of certain carbonates and silicates of the alkaline earths and earths, as well as in these earths themselves, and in zinc and tin oxides; *changes color* permanently or transiently, the following being some of the most characteristic changes, which may, however, be very numerous; from *white to transient yellow,* zinc oxide [5] and many of its salts; *white to permanent yellow,* reddish while hot and fusible at high temperature, white compounds of *lead* [6] and *bismuth oxides; tin binoxide* is infusible and remains pale yellow on cooling; *reddish to transient black, ferric oxide; brown to transient black,* reddish on cooling, hydrous ferric oxides, as *limonite; pale yellowish-brown or grayish to black, siderite** [8]; *green, blue or red to black, salts of copper* [7] and *nickel* and *cobalt; yellow to dark orange,* fusible at high temperature, *potassium chromate.*

b. Charring indicates organic compounds and is generally accompanied by an odor of burnt sugar or a bituminous odor (in presence of nitrogenous matter the odor often resembles that of burning

* The black residue left by siderite is magnetic oxide of iron; magnetic residues are also left by some other iron compounds which undergo partial decomposition, as pyrite, § 31, 2, *a*, etc.

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feathers). Fumes are often evolved which condense on the sides of the tube to an oily liquid [9], having an acid or alkaline reaction. Effervescence of the charred residue may indicate organic salts of the alkalies, especially if the filtered aqueous solution of any soluble matter in the residue effervesces with acids, with escape of carbon dioxide. Blackening alone does not necessarily indicate *charring* (but if the black residue takes fire or glows until consumed, when heated in the open air, the presence of carbon may be assumed. —FRESENIUS, New York, 1880).

c. Gases or fumes are evolved.

1. **Oxygen,** which rekindles, or at least causes to glow more brightly, a splinter of wood or charcoal, introduced into the tube while a spark still glows on its end; it is given off by certain chlorates, bromates, iodates and oxides [2]. The manganese binoxide should be powdered and a somewhat large quantity employed, with a rather high temperature, the top of the tube being loosely closed with the finger to prevent the loss of the gaseous oxygen.

2. **Ammonia,** recognized by its odor, or its effect on test-paper, § 61, is given off by some ammonium salts, and sometimes by nitrogenous organic bodies [9], when charring usually ensues; compare *b*.

3. **Iodine,** in violet vapors, from certain iodates and iodides of weak bases and free iodine.

4. **Nitrogen oxides** (trioxide and tetroxide) in brownish-red or yellowish-red fumes, yielded by certain nitrates [10] and nitrites. If in small quantity the color of such vapors is best seen by looking lengthwise through the tube held over a white surface.

Bromine sometimes escapes in similar colored fumes from certain of its compounds.

5. **Cyanogen,** from certain cyanides, is recognized by its odor. It burns with a crimson flame, if pure enough.

6. **Carbon dioxide,** (colorless and practically odorless if pure) from carbonates decomposed by heat, oxalates, and possibly from partial oxidation of carbonaceous matter. It renders a drop of lime-water (made by digesting strongly ignited calcium carbonate, or calcite, in water and filtering after some time) turbid when exposed to the gas on the end of a glass rod or on a watch-glass.

7. **Carbon monoxide,** colorless, practically odorless, burning with a pale blue flame if pure, is yielded by formates, which blacken, and by oxalates, in which case it usually contains carbon dioxide also.

8. **Hydrosulphuric acid,** readily known by its characteristic odor, is yielded by sulphides containing water; also by some sulphates, in presence of reducing matter and hydrous bodies yielding water at a high

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temperature. It is also easily detected by holding paper slightly moistened with lead acetate solution at the mouth of the tube, when a dark brown or blackish stain of lead sulphide will form on the paper.

9. **Acid fumes,** yielded especially by sulphites and sulphates of weak bases. The fumes redden blue litmus paper, or condense, with any water expelled from the substance, to drops on the inside of the tube, which have an acid reaction.

10. **Hydrofluoric acid,** from fluorides of weak bases containing water, attacks the glass and is also identified by means of Brazil-wood paper.

d. Water, condensing more or less abundantly on the sides of the tube, may be mechanically combined with the substance, or may come from hydrated oxides, or salts containing basic water, or water of crystallization, often forming in the last case large drops. It should be tried with test paper, and may be neutral [11], alkaline or acid, the acids of *c* being sometimes also detected.

31. *A sublimate forms, i. e.,* the vapors condense to a *solid* within the tube when cold (mercury remains *fluid,* manifestly so if in any considerable quantity).

The most important are

1. **White sublimates.**

a. Ammonium salts,
b. Mercurous chloride [13],
c. Arsenous oxide [14], a sublimate of octahedral crystals.

} Sublime without fusing.

d. Mercuric chloride, fuses before subliming.

e. Antimonous oxide, fuses to a yellow fluid and sublimes in lustrous needles, if the air is excluded.

f. Tellurous oxide, fuses, and at a high temperature yields an amorphous sublimate.

2. **Colored, Black, Gray, or Metallic Sublimates.**

a. Dark yellow to *brownish-red* while hot, *yellow* on cooling.

Sulphur, pure *sulphur-yellow* on cooling; fuses to distinct clear drops or a continuous coating if abundant; indicates the presence of free sulphur or sulphides of the heavy metals containing much sulphur, like *pyrite* [15], *chalcopyrite, hauerite,* etc.

[*Mercuric iodide* gives a fusible red to yellow sublimate, bright yellow and crystalline on cooling (bright scarlet also if abundant) becoming red when rubbed.]

b. Brownish-red (to almost *black* if abundant) while hot, *reddish-yellow* to red on cooling, *Sulphide of arsenic,* present as such [16],

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or formed from compounds of metallic sulphides and arsenides. Compare *f*.

c. Black while hot, *brownish-red* when cold. *Sulphide* with *oxide of antimony*, indicating the sulphide, either alone [17], or with other sulphides; *berthierite, tetrahedrite, bournonite.*

d. Dull black, giving a red powder when scraped out and rubbed on paper. *Sulphide of mercury*, artificial or native [18]; also minerals containing mercury and sulphur with other metals, *mercuriferous tetrahedrite.*

e. Dark red, nearly *black*, accompanied by an odor of decaying horse-radish. *Selenium*, indicating *selenides. Tiemannite* [19] gives a dark sublimate with reddish border.

f. Lustrous, black, metallic; crystalline when abundant. *Metallic arsenic*, existing as such [20], or set free from certain arsenides; *smaltite* [21], *arsenopyrite* [22]. In case of this last mineral the arsenic sublimate is preceded by the sublimate *b*.

g. Lustrous, metallic, grayish-white mirror-like film. *Mercury*, either originally free, or liberated from the oxide [23] or amalgams, § 84, *b*. Unless rather abundant, the mercury appears as a grayish-white film, which the magnifying glass shows to consist of minute globules or dots of shining mercury.

h. Lustrous dark gray or *black, metallic mirror*, near the assay and formed at a rather high temperature. *Cadmium* [24] and certain of its alloys, or its oxidized compounds [25], if the latter are mixed with a reducing agent like coal dust. The sublimate shows more or less of a brown border; see also § 91, *b*.

i. Lustrous metallic globules, forming at a very high temperature. *Tellurides.*

B. Examination in the Open Tube.

32. Place a bit of the substance, not larger than a hemp-seed, a little within the lower end of the tube, § 8, *a*, and heat this end over the alcohol flame, or with the O. F., at first very gently, then to full redness. The tube must be inclined so as to produce an upward current of air through it, and the flame must not enter the tube. If no result is obtained from the fragment, treat a fresh portion of the substance in powder. The effect in the open tube is oxidizing.

Substances that fly to pieces when heated should first be powdered. A very narrow strip of moistened blue litmus paper is to be inserted in the upper end of the tube when testing for sulphur, and too high a heat must not be applied at first, since very volatile sulphides, as of arsenic and mercury, may pass off unoxidized, yielding a colored

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sublimate where only a white sublimate of arsenous acid, or a mercury sublimate, should have been obtained.

1. **Odors.**

a. Like burning *sulphur*, caused by fumes of sulphur dioxide (commonly called sulphurous acid), which redden the litmus paper, indicating: *Free sulphur, sulphides* [15], *sulphites* and *sulphates of weak bases.*

b. Like *decaying horse-radish*, disagreeable and highly characteristic, indicating: *selenium* and *selenides* [19], compare 3*a* below.

c. A *garlic* odor, especially perceptible when the tube is somewhat rapidly heated, is very characteristic of *arsenic* [20], compare 2*a*.

[*d. Osmium tetroxide*, having a very irritating and characteristic odor, is readily obtained from finely divided osmium and platinum residues containing osmium.]

2. **White Sublimates.**

a. Crystalline (best observed with a lens), easily driven about by gently heating the tube: *Arsenous oxide* free [14], or from *arsenic* [20], *sulphide of arsenic* [16], or *arsenides* [21]. A dark or colored sublimate may form with the latter if too quickly heated, see 4*b*.*

b. Frequently preceded by *heavy white fumes* and partly condensing at first on the upper part of the tube, but afterwards in a denser sublimate on the lower side. The former can be for the most part driven off by a red heat and consists mainly of *antimonous oxide;* the latter is chiefly a non-volatile, infusible compound of *antimonic* and *antimonous oxides*, yellowish white while hot [17].

Less characteristic and not so important are the following:

c. Non-volatile sublimate, condensing on the lower side of the tube, fusing to drops, yellow while hot. *Sulphate of lead*, indicating *sulphide of lead.* (*Sulphide of bismuth* does nearly the same, but the sublimate remains yellowish on cooling.)

d. Scanty, partly crystalline, forming with difficulty and fusing to drops, yellowish while hot. *Molybdenum trioxide*, free, or from *sulphide of molybdenum.* Touched directly by the R. F., becomes blue or reddish.

e. Partly volatile, fusible to drops, yellow while hot, pearl-gray to white when cold. *Oxychloride of lead*, indicating *chloride of lead.*

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* The light-blue flame-coloration of arsenic, § 42, 5, *c*, can be well observed by heating the open glass tube so that the white sublimate of arsenous oxide will escape from its upper end, and holding this end in the alcohol flame. The same method brings out clearly the antimony flame-coloration, § 42, 4, *e*.

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f. White fumes, condensing to a grayish sublimate, fusible to colorless drops. *Tellurous oxide*, from tellurium and tellurides.

3. **Steel-gray and Metallic Sublimates.**

a. Dark steel-gray near the assay, generally with reddish edge above, and sometimes with small, very volatile crystals of white *selenous oxide* beyond this, indicates *selenium* or *selenides* [19], see 1, *b*.

b. Lustrous, gray, metallic mirror, sometimes only a very scanty deposit of lustrous globules, distinctly visible under the lens, in reflected light, indicates *mercury*, which may be collected into drops by rubbing, if abundant, and comes from *sulphides* [18], *amalgams* or *oxide of mercury*, see § 84.

4. **Colored Sublimates.**

a. Brown sublimate very near the assay, yellow on cooling and fusible to drops, yellow when cold. *Oxide of bismuth*, from compounds of *metallic bismuth*.

b. Yellow and *red sublimates, sulphides of arsenic ;* reddish, *sulphide of antimony ;* dull black, *sulphide* of mercury ; they all result from too rapid heating (see introductory remarks of § 32).

C. Examination on Charcoal Alone.

The student should carefully read §§ 6 and 7.

General Remarks.

33. Substances are treated on charcoal either alone or with appropriate reagents. Occasionally it is desirable to submit a fragment of some substance to a high temperature on coal, and then difficulty may be found in keeping it from being driven off by the blast. This difficulty may often be avoided, as suggested by Brush, by causing the fragment to adhere to a *very small* bit of borax, previously fused on the coal, but this method must be used with caution, since, for instance, a very little borax may materially affect the fusibility of many compounds with which it comes in contact.

Apart from the important coats, presently to be described, § 34, and other phenomena reserved for special description, §§ 40, 41, 42, the following points are to be noted in the treatment on coal alone.

a. Volatilization, entire or partial, with or without formation of a coat on the coal [13], [22].

b. Fusion, the substance also sinking into the coal ; as do the caustic alkalies and many of their salts [49] and [26], forming at the same time more or less of a coat ; also barium and strontium oxides and barium carbonate, after its carbon dioxide has been expelled by prolonged heating. (Compare § 54, *b*.)

c. Change of color, very much as in the closed tube, § 30, *a*.

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d. Intense glowing, as in case of lime, magnesia, zirconia and other earths and their infusible salts, when highly heated. Zinc oxide [28] and tin binoxide show also a phosphorescent light, even when less highly heated, § 35, *c, d.*

e. Deflagration, more or less violent, indicates nitrates, chlorates, bromates and iodates.

f. Alkalinity, § 41, shown by many neutral salts of the alkalies and by the carbonates, nitrates, sulphates, chlorides, fluorides and several other salts of the alkaline earths.

g. Magnetism, shown in R. F. by certain salts and the oxides of iron [57] nickel and cobalt, which are reduced to metal (or, in the case of iron, only to the magnetic oxide). In O. F. also certain iron salts [15] yield magnetic residues if not too thoroughly oxidized.

h. Decrepitation and *evolution of gases* will occur, much as in the closed and open tubes, §§ 30, 32.

i. Reduction to metal (compare § 50). Oxides and many salts of the following metals. Gold and copper, yielding fusible, malleable globules and no coat. Rhodium, ruthenium, palladium, iridium and platinum (all reducing easily), nickel and cobalt (with difficulty), yield infusible metallic powders or masses and no coat. Molybdenum trioxide reduces with great difficulty to metallic powder and yields a coat. Silver reduces easily from most of its compounds, yielding malleable, fusible globules and little coat unless highly heated. Antimony, bismuth, lead and tin (this with difficulty) are reduced to fusible globules and yield more or less abundant coats. Antimony gives brittle globules, bismuth rather so.

COATS FORMED ON CHARCOAL ALONE.

34. The coats formed under different conditions by various elements and compounds will be here described. The student should study them carefully, and he will then be able, with the help of the Table, § 39, to refer any similar phenomena to their true causes (compare § 53).

For the preliminary test on coal place the substance alone in a very shallow cavity or depression, merely scraped on the coal and not bored deep, so as to present sharp edges, which would deflect the passing flame upward from the coal. The depression should serve only to keep the substance from rolling off, and should be quite near the end of the coal, so that it can readily be covered by either O. F. or R. F. Hold the coal horizontally, at a right angle with the lamp, and direct the flame down on the assay piece at a moderate angle, so that the current of the blast may sweep over it, carrying along any escaping

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fumes, and depositing them, if they condense, in a longer or shorter coat on the face of the coal, not in a ring around the assay, but mainly on the farther side of it. Students often may lose most of the coats by directing the flame *across* the coal instead of along its length. Where a rather volatile coat may be expected a long coal must be used, or the sublimate may escape unnoticed. The O. F. and R. F. must be used as directed, and to obtain a good coat use a moderately strong R. F., unless the O. F. is especially mentioned. Powders will frequently blow away, unless pressed down on the coal and very gently heated at first. Sometimes they must be moistened. Little attention need be paid, in actual practice, to the unimportant reactions enclosed in brackets, although they must not be entirely passed over, being given here to notify the student that they may occur, while yet they are not characteristic. All the other tests must be carefully performed and the phenomena noted. The colored flames obtained will be described in § 42.

Note.—Observe first the color of the ash yielded by the coal alone. It should be light gray or white; a brown ash may mislead the operator. Generally the coal ash can be very easily blown away, while white coats, excepting sometimes oxide of tin, remain quite fast. A white coat may appear bluish-white if very thin.

Substances yielding White Coats especially.

(Some of these coats may be more or less yellow while hot.)

35. *a. Arsenic* [20]. The metal and its oxides [14] volatilize in both flames without fusing, and yield a very volatile white coat of arsenous oxide, far from the assay and quickly disappearing when heated before the tip of the O. F., although the coal be held far from the flame and by no means brought to glowing. If quite copious and quickly heated with the inner flame, p. 30, this coat can generally be made to impart a distinct light-blue color to the flame, and the same coloration is obtained by heating the original substance in the inner flame. When the assay is heated for only a short time in R. F. and held quickly under the nose, it yields a peculiar, alliaceous odor. (If very abundant fumes are given off they should not be too freely inhaled; see § 97, *a.*)

[*Note.*—The *sulphides of arsenic*, too rapidly heated on coal, may volatilize without being completely oxidized, and may then form yellowish or reddish coats, instead of the white coat of arsenous oxide.]

b. Antimony [29]. The metal fuses and volatilizes easily in both flames; the oxides [30] best in R. F., being partly reduced to shining globules of the metal. All tinge the inner flame pale yellowish-green (to be very carefully noted) and yield a white coat of oxide,

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rather nearer the assay than that of arsenic, and easily volatile, but much less so than the arsenic coat. [With S. Ph. and tin this coat gives a dark bead, § 96, *b*. Omit this last test at present.]

The metal, fused a moment in R. F. and removed from the flame, continues in fusion, yielding copious white fumes and becoming rapidly surrounded with very slender crystals of antimonous oxide.

[*Sulphides of antimony*, too rapidly heated in strong R. F., may yield a slight reddish coat; see remarks at end of *a*, under sulphides of arsenic.]

c. Zinc [31]. The metal fuses easily in either flame, volatilizes in vapor which burns with a very characteristic, whitish-green flame and yields an abundant coat, while flakes of white oxide often rise in the air. The coat is best obtained in R. F. and is yellow while hot, white when cold. Under the O. F. the heaviest parts of it glow with a greenish-yellow, characteristic phosphorescence, but remain fixed. This coat, as well as the oxide [5] and all reducible compounds of zinc, is slowly reduced to metal in R. F. and this volatilizes, yielding a new coat of the oxide, quite like that described above.

d. Tin. The metal [32] fuses easily in either flame, but before a gentle O. F. is quickly covered with a non-volatile, infusible coat of oxide of tin. Heated just within the point of the inner flame, the metal remains bright and gradually yields a copious coat of oxide, beginning very near the assay, dirty yellowish-white while hot, white when cold, and becoming slightly luminous in O. F. It may, for the most part, be easily blown from the coal, and volatilizes in R. F. only with difficulty.

The oxide [33] and other reducible compounds of tin are reduced with some difficulty by a strong R. F., the assay being put in a deeper cavity for the sake of securing better reducing action, § 7. A part of the metal after being reduced is again volatilized and undergoes oxidation in the air, forming anew a little of the oxide coat, as obtained from the metal itself [32] above, while the greater part of the reduced tin can be seen in minute globules in the cavity. The color and white, metallic lustre of the tin thus reduced is best observed by scraping these globules and the coal around them into a mortar, washing them free from the coal dust, § 51, and flattening the tin globules in the mortar by gently rubbing them with the pestle. This reduction of the oxide will require a very good and powerful R. F.

e. Tellurium. Fuses easily in both flames and yields a white coat of tellurous oxide, with a red or dark yellow border. The coat is volatilized easily by either flame, and tinges the inner flame green.

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f. Thallium. Fuses easily, yielding a very volatile white coat, sometimes brown near the assay, and imparting an intense green color to the inner flame.

g. Molybdenum. The metal is infusible. Its trioxide, commonly called *molybdic acid* [1], fuses in O. F. and volatilizes, yielding a yellowish coat, white on cooling. If the R. F. is brushed rapidly across this coat it leaves a deep blue mark, but if the coat is strongly heated in O. F. it partly volatilizes, while there remains a very characteristic, copper-red film of binoxide of molybdenum.

36. [*White coats*, in general less characteristic than the above, are frequently obtained, which demand attention because they may lead to mistakes and also because they afford useful indications in case of simpler substances, although the compounds affording them cannot always be thus easily detected when in mixtures, even by experienced operators.

They are yielded by:—

a. Chlorides, bromides, iodides, sulphides and *sulphates* (these latter being reduced to sulphides and then volatilizing and again forming sulphates) of *potassium, rubidium,* and *caesium,* coloring the inner flame violet; of *sodium,* coloring it yellow, and of *lithium,* coloring it carmine-red.

b. Chlorides of ammonium, mercury, and *antimony* (which colors the inner flame pale yellowish or bluish-green before it volatilizes, if in large quantity) and *chloride* and *iodide* of *cadmium* yield white coats that do not color the flame.

c. The white coats formed by *chlorides of lead, bismuth, zinc* and *tin, iodide of zinc,* and the *sulphides of lead* and *bismuth* (which yield white coats of their sulphates) are accompanied by the less volatile, characteristic coats of the oxides of these metals, and the *chloride of lead* coat colors the flame blue; compare § 86, *a.* The white coats so formed by lead and bismuth sulphides leave a yellow spot when heated with the point of the flame until the coal glows (distinction from antimony and arsenic) and the lead coat tinges the inner flame blue.]

Yellow Coats.

37. *a. Lead* [34]. The metal fuses easily in both flames, while its oxides, their salts [6] and other reducible lead compounds are readily reduced in either flame. All yield a coat of oxide (due to oxidation of the volatilizing lead), dark lemon-yellow while hot, paler sulphur-yellow on cooling, and often having a more remote border of white carbonate of lead, bluish-white in thin layers and not characteristic. If the coal is heated to glowing the coat is volatilized, because reduced to metal gradually even in O. F., and tinges the inner flame characteristically faint *azure blue.* If the white coat of the *carbonate* or of the *chlorides, etc., of lead,* § 36, *c,* be treated with the point of the O. F., until the coal glows, it partly volatilizes, but leaves also a yellow spot of oxide.

b. Bismuth [35] and its compounds [36] behave quite like lead,

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but the coat is dark orange-yellow while hot, lemon-yellow on cooling, and there is no coloration of the flame. [With S. Ph. and tin the coat gives a characteristic reaction, in common with antimony, § 90, *a*; omit this for the present.] Compare also § 36, *c*.

[*c*. *Cadmium* when present in small quantities may yield mainly an orange-yellow coat. See also § 38, *a*.]

d. *Indium*. The metal fuses very easily, volatilizes and burns in O. F., tinges the inner flame a deep purplish-violet and yields a coat of the oxide, dark yellow while hot, light yellow on cooling, and volatilizing slowly in R. F. with its characteristic flame.

[*e*. Iodides of *lead*, *bismuth* and *mercury* may yield yellow or red coats.]

[*f*. Silver may afford a slight yellowish-brown coat, § 38, *b*.]

Brown, Red, and Gray Coats.

38. *a*. *Cadmium*. The metal [24] fuses very easily in both flames, taking fire in O. F., and emits copious, dark yellowish-brown fumes, which form a very characteristic coat, varying from dark reddish-brown near the assay to orange-yellow at a greater distance, and usually having more of the latter color when less abundant. It is easily volatilized even in O. F., being reduced to metal. A characteristic iridescence is generally visible on the farther edge of the coat, especially when somewhat slowly formed. There may sometimes also be formed a very dark or even crystalline coat, quite near the metal.

The *oxide* [25] and all cadmium compounds which can be reduced to metal yield the above coats, but somewhat less readily.

b. *Silver* [37]. Fuses easily and remains bright in both flames; under a strong O. F. it may be slowly volatilized, so as to form a slight yellowish-brown to brownish coat of oxide, too obscure to be a good test. If a very little lead or bismuth is present, a yellow coat will form, § 37, *a*, *b*, followed after longer blowing by a characteristic red coat; if a little antimony is present the coat is first white, § 35, *b*, and after longer treatment, red.

Silver oxide [38] reduces readily to the metal in either flame, and the metal thus obtained finally yields the same brownish coat.

c. *Selenium* fuses easily in either flame and yields brown fumes and the characteristic, disagreeable odor of decaying horse-radish. Near the assay is deposited a steel-gray, metallic, feebly lustrous coat, which is dull, dark gray farther away, with sometimes a reddish border. It volatilizes in both flames, and tinges the inner flame azure blue. *Tiemannite* [19] may be used for obtaining the most characteristic reactions.

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[*d. Chloride of copper* [39] volatilizes in part, undergoing partial decomposition, coloring the inner flame beautiful azure-blue and yielding white fumes, with an odor of chlorine. A yellow to brownish coat also forms, with a gray one nearer the assay and sometimes a bluish-white one on the outer edge.]

[*e. Oxide of mercury* and substances containing much metallic mercury may yield a grayish coat of this metal on coal, very volatile and at a distance from the assay.]

TABLE OF COATS ON CHARCOAL.

39. The colors refer to the cold coat, unless otherwise stated, and no reagents are supposed to have been used, or only (as regards subsequent references) simple reducing ones, like soda, potassium cyanide or potassium oxalate.

White:

Arsenous oxide; in R. F., arsenic odor; very volatile; tinges inner flame blue; leaves no yellow spot. § 35, *a.*

Antimonous oxide; quite volatile; tinges inner flame pale, yellowish-green; leaves no yellow spot. § 35, *b.*

Zinc oxide; yellow while hot; not volatile in O. F., but slowly in R. F. § 35, *c.*

Tin oxide; pale yellow while hot; not volatile in O. F. and but slowly in a strong R. F. § 35, *d.*

Tellurous oxide; volatile; tinges inner flame green. § 35, *e.*

Thallium oxide; volatile; tinges inner flame intense green. § 35, *f.*

Molybdenum trioxide; yellow while hot; after brief treatment in R. F., blue; under strong O. F. a red film. § 35, *g.*

[*Various chlorides, sulphates, etc.*, § 36, *a, b, c.* If the white coat, strongly heated in O. F., leaves a yellow spot, lead or bismuth compounds. § 36, *c;* § 37, *a, b.*]

Yellow:

Lead oxide; gradually volatilized even by O. F., if strongly heated; tinges inner flame blue. § 37, *a.*

Bismuth oxide, like lead oxide, but somewhat darker and not imparting a blue color to the flame. § 37, *b.*

Cadmium oxide; (also, if abundant, dark, or reddish-brown, in part;) easily volatile in either flame; iridescent border. § 37, *c*, and § 38, *a.*

Indium oxide; tinges inner flame purplish violet. § 37, *d.*

[*Iodides of lead and mercury.* § 37, *e.*]

[*Chloride of copper.* The assay colors the R. F. intense blue, § 38, *d.*]

[*Silver oxide.* § 37, *f.*]

Brown:

Cadmium oxide; if abundant, dark nearer the assay, yellow at most distant border (see above under yellow coats). § 38, *a.*

Silver oxide; indistinct and only after prolonged treatment with a strong O. F. § 38, *b.*

[*Chloride of copper.* The assay colors the inner flame intense blue. § 38, *d.*]

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Red:
- *Silver with lead;* generally preceded by the yellow lead oxide coat. § 38, *b*.
- *Silver with antimony;* preceded by the white oxide of antimony coat. § 38, *b*.
- [*Iodides of bismuth and mercury.* § 37, *e*.]

Gray:
- *Selenium;* odor of horse-radish also evolved. § 38, *e*.
- *Arsenic suboxide;* with white coat of arsenous oxide (compare arsenous oxide, above). § 35, *a*.
- [*Chloride of copper;* (compare above, under brown and yellow coats). § 38, *d*.]
- [*Mercury*, § 38, *e*.]

D. Examination for Fusibility.

40. Solid substances are tested in the platinum forceps, § 10, *a;* the sharp, thin point of a very slender fragment being held for this purpose edgewise in the tip of a very hot O. F., at *d*, Fig. 21, but not in contact with the blue cone if they are reducible; compare § 26. Take care in general not to let any of the fused substance come in contact with the platinum, which would probably be injured if metallic sulphides or metals were under treatment. Compare note, § 9. Even powders may sometimes be tested in the forceps if they are first mixed with water to a stiff paste, which is spread thinly on coal and dried with a very gentle O. F., being heated just enough to become hardened to a crust, which is then lifted off and tested in the forceps; otherwise they must be tested on coal. Substances that decrepitate or fly to pieces before the flame, are first heated in a closed tube or matrass, until they cease to decrepitate, and the resulting powder is treated as above, unless a fragment suitable for the forceps can then be found. In case of metals and substances generally that have a metallic lustre, the platinum may be injured, unless great care is taken. In such cases the student will do well to test the fusibility on coal first, taking care to avoid the effect of reducing gases as much as possible.

Always use the O. F. for testing the fusibility of bodies, since a reducing action may alter them, as in case of the infusible sesquioxide of iron, which becomes slightly fusible when reduced to the proto-sesquioxide. When a substance fuses with very great difficulty use the finest splinters, since some substances only become rounded on the thinnest edges in the hottest O. F., and such should be examined with the magnifying glass after treatment.

Observe also whether the substance bubbles and swells (*intumesces*) [40]; shoots out little sprouts (ramifies) [41]; swells and curls up (*exfoliates*) [42], with or without fusing; whether the resulting

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fused mass is glassy and quite clear [43]; enamel-like [4]; rendered more or less opaque by numerous very small bubbles, forming a *blebby* or *bubbly* glass, or is only partly fused to a vesicular scoria; all of these being very important in distinguishing minerals.

The following scale of fusibility, by Von Kobell, is generally adopted:

1. *Stibnite* [17], coarse splinters fuse in a candle flame.
2. *Natrolite* [43], fine splinters fuse in a candle flame.
3. *Almandite* [44], (iron-alumina garnet), coarse splinters fuse easily B.B.; infusible in candle flame.
4. *Actinolite* [45], less readily fusible in coarse splinters B.B.
5. *Orthoclase* [46], B.B. fusible in very fine splinters.
6. *Bronzite* [47], B.B. only rounded on the edges of the finest splinters.

E. Examination for Alkalinity.

41. Certain salts of the alkalies and alkaline earths acquire an alkaline reaction after ignition, although not reacting alkaline before. The ignition is best effected in the platinum forceps in O. F., but on wire or coal if necessary, as directed in § 40 for fusibility. After cooling, the ignited substance [27] is placed on slightly moistened turmeric paper, which it will turn reddish-brown, or on red litmus paper, which it will turn blue, § 16, 2, 3.

F. Examination for Flame Coloration.

42. A moderately strong O. F. is produced, the blue inner cone of which, p. 30, must be perfectly free from streaks of yellow flame and not too pointed, and the substance must be held just within the tip of the *inner flame*, the coloration of the flame being much feebler, or not appearing at all, if the substance is held beyond this in the true O. F. Fragments are held in the platinum forceps, with due caution, § 40, and *Note*, § 9. If very fusible, or if in powder; the substance is tested on platinum wire, or if likely to injure platinum, it is laid in a shallow cavity on charcoal, and brought within the inner flame. The inner flame has a reducing action, hence all compounds of easily reducible metals might injure platinum. Substances which decrepitate strongly are treated in powder on wire, or else are prepared as in § 40. Pure water must always be used when substances are to be moistened, and the platinum wire first tested alone, to see that it gives no color. The alcohol flame from a clean wick is good for these tests, especially when made on charcoal, but the gas flame

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is best of all. The oil lamp is available, however. The colored flames are best seen in a somewhat dark room, or against a dark background, formed by pieces of charcoal or the like, placed behind the lamp. A fresh portion of the substance is generally taken for each test. Sometimes the assay must be moistened with a very little concentrated sulphuric acid, in which case the coloration is often so transient as to require the closest attention; in many cases the addition of a drop of hydrochloric acid, to form chlorides, is useful, especially with sulphates after first heating in R. F. Remember that the substance must be brought just within the blue inner flame, and a very little of any soda salt may conceal the reaction entirely. The fingers pressed on the wire or substance will usually cause a yellow soda flame.

The following colored flames are characteristic: for the modifications produced by mixtures consult the special reactions for each element. The colors often come out strongly only after prolonged heating. Sodium compounds very often occur as impurities with other salts, and these conceal the true coloration the latter would impart to the flame. The student, therefore, before concluding that the substance will yield only the yellow sodium flame, must heat it a long time, noticing whether at last, while some of the substance remains, the sodium flame disappears wholly or in part, giving place, perhaps, to some other coloration. Lastly, the coloration is generally better obtained from rather small quantities, which can be more highly heated.*

1. Yellow.

Sodium compounds [26] give a more or less intense yellow color, with a very slight reddish tinge. The test is exceedingly delicate.

2. Violet.

Potassium compounds [49] generally, except the borate and phosphate, give a bluish-violet color, readily masked by the sodium or lithium flames, see § 56. The very rare *rubidium* and *caesium* compounds behave much like the potassium salts. *Indium* compounds give a fine purplish-violet flame. Indium is found only in exceedingly small quantities in a few minerals, and never in such proportion that the raw mineral can give the indium flame.

* The substances here cited as examples may be tested on platinum wire, unless otherwise directed.

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3. Red.

a. Carmine. *Lithium* compounds [50]; test in the platinum forceps.

b. Red. Most compounds of *Strontium* [41]; in the forceps. The chloride gives a very intense red, sometimes resembling the lithium flame; the color, from minerals, if faint, is intensified by moistening the assay with hydrochloric acid, after first igniting it strongly, best in the point of the blue cone of the inner flame. Barium compounds readily conceal the strontium flame.

c. Yellowish-red. *Calcium* (*lime*) compounds generally [27] (in the forceps), except silicates, of which wollastonite alone gives a good lime coloration. The flame from fusible calcium salts is often mistaken for a strontium coloration by beginners, and direct comparison of the two should be made when there is any doubt. Most lime minerals require prolonged heating and the sulphates of lime do not give a strong coloration.

4. Green.

a. Emerald-green. Most compounds of *copper* (and the metal itself, strongly heated on coal). *Copper iodide* gives a very intense green flame.

b. Green. *Thallium* compounds (intense green). *Tellurous acid.*

c. Yellowish-green. *Barium* compounds (except silicates). Stronger after prolonged heating, and very persistent [51], in powder on wire.

Oxides and *sulphide of molybdenum.*

Boric (*boracic*) *acid* [52] (on wire) and *borates*, the latter especially with sulphuric acid on platinum wire, when the color is often very transient, ceasing as soon as the sulphuric acid has evaporated.

d. Bluish-green. *Phosphoric acid* and *phosphates*, some only in powder with sulphuric acid [53] (on wire) and then transient. This flame can be well seen around a match flame, before the wood ignites.

e. Pale yellowish-green to bluish-green. *Antimonous oxide* [30], or *antimony* and its compounds on coal. This flame must be carefully observed.

f. Whitish-green. *Zinc* [31] on coal. [In a powerful R. F. this coloration may sometimes be obtained from zinc oxide.]

[*Note.*—Certain *ammonium* salts with *nitric* and *hydrochloric acid* yield a feeble greenish flame, not used as a characteristic test.]

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5. Blue.

a. Azure-blue. Selenium (intense), [19], on coal.

Lead [34] (on coal) and its compounds; the *phosphate, pyromorphite*, giving a blue flame, faintly tipped with green (in the forceps).

Copper chloride [39] (on coal); a very intense and characteristic blue flame, afterward changing to green. The blue flame is given by other copper compounds and the metal, after being moistened with hydrochloric acid.

b. Greenish-blue. Copper bromide; intense, somewhat like the *chloride;* soon changing to green.

c. Light-blue. Arsenic [20] (on coal) and its compounds, in absence generally of other coloring bodies.

G. Roasting.

43. *Sulphides,* identified by § 103, *b,* and *arsenides** (in certain cases also *arsenates*) of the heavy metals, including antimony, must be thoroughly roasted before being treated with borax or S. Ph., often also before being tested with soda, unless for especial reasons. The roasting brings the metals, for the most part, to the state of oxides.

In a very shallow, wide cavity on a piece of charcoal, spread a thin layer of the finely powdered substance, pressing it down gently with the spatula, and heat with the O. F., at first very little, to avoid fusing it, and then at a low red heat, until no further odor of sulphurous oxide (or acid), or escaping fumes of arsenous oxide are perceptible. Then treat the powder for a short time with the R. F. to reduce arsenates, or sulphates, if present, until any arsenic odor ceases, and repeat the treatment with O. F. and R. F. alternately, until no more fumes of sulphurous or arsenous oxide escape. The powder will then generally cohere to a crust, which is carefully turned over with the knife point and treated again with O. F. and R. F. alternately as before. Sulphides will often be sufficiently roasted by one careful operation, but arsenides generally require to be again finely powdered and roasted, especially when nickel or cobalt is present.

Selenides, tellurides, and antimonides with little sulphur need no roasting.

Some sulphides, especially of antimony or lead, with copper and

* In special cases of treatment on coal arsenides are not first roasted; compare particularly § 76, *d.*

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other metals, fuse very readily, and do not then roast easily. When such substances require roasting, they should first be fused on coal, until a part of the antimony, etc., is volatilized, and the resulting mass powdered and thoroughly roasted. A substance after being properly roasted will, unless reduced to metal in the operation, have a dull appearance and be easily rubbed to a powder. If it is desirable to prevent too much arsenic fumes from escaping, the substance can first be freed from the greater part of the arsenic by heating in the closed tube.

H. Examination with the Glass Fluxes.

General remarks on the use of the glass fluxes.

44. Borax and salt of phosphorus (S. Ph.), are called glass fluxes because they melt to glassy beads, transparent when cold. Compare § 12, 1, 2.

1. General Remarks on the Borax Bead.

a. Making the borax bead. This is made by heating the platinum wire loop, § 9, *a*, in O. F. and touching it quickly to a bit of granulated borax glass of proper size (or to powdered borax), after which the borax is fused in a strong O. F. to a bead. This must be perfectly fused, almost or quite free from bubbles, colorless even when hot, § 74, *a*, *Note*, and perfectly clear both when hot and cold. It must be of suitable size, and not too thin, but nearly spherical. When powdered borax is used, the wire will often have to be again heated and dipped into it, in order to get a bead of good shape.

Impure borax sometimes gives a cloudy or opaque bead when cold, but generally any such appearance is due to imperfect fusion. Substances are brought into the bead simply by touching it, while still hot, against them, and then fusing the bead again to dissolve the substance. It may here be stated that metals do not dissolve in the fluxes; it is the oxides of the metals that dissolve.

In general the O. F. should be used to make the bead and to dissolve in it the substance to be tested, unless otherwise directed, no matter whether the O. F. or R. F. is to be used in the subsequent examination. This is done partly because the O. F. is hotter than the R. F., and partly because a premature reduction may sometimes do harm. When the student knows that a partially reducing flame can do no harm in the first solution of the substance in the flux, he may at once bring the bead just up to the tip of the inner flame, for here the heat is greatest and solution is soonest effected.

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The student will do well to avoid working with very large beads at first.

b. It is often necessary to treat substances beside borax beads on coal. In this case a bit of borax glass or a borax bead is placed beside the substance, in a very shallow cavity, if oxidation is desired; in a deeper one if the R. F. is to be used. Occasionally the powdered substance is mixed at once with the powdered flux (best the powdered borax glass) and the mixture melted on coal. Almost invariably it is desirable to get the melted borax into a spherical bead and not leave it spread over the coal. The proper way to accomplish this, under certain circumstances, is described in *d.*

c. It is sometimes desirable first to form a bead on wire and afterward treat it on coal. When this is done the bead is best detached from the wire by fusing it until it is quite fluid and then detaching it by striking the ball of the thumb smartly against the table, the wire loop being first brought close over a porcelain dish into which the bead may fall.

d. Certain oxides cannot be reduced from borax B.B. alone, and can therefore be readily separated from the reducible ones. The operation is modified according to circumstances. If the oxides have been already dissolved in borax on wire in O. F., as in *c*, or on coal, the borax bead is next treated with a strong R. F. in a moderately deep cavity on coal. For this purpose a charcoal crucible, § 138, *b*, is most convenient; but if the hole is bored in the end of a sound and not too thick piece of charcoal, this serves nearly as well. It is not so good a plan to make the hole simply in one of the side faces of the coal, because this arrangement does not so readily admit of the motions necessary to bring the collecting metal, presently to be mentioned, into contact with all parts of the melted borax.

Unless there is enough reducible and fusible metal to be furnished by the oxides treated, a bit of gold, silver, or lead is added to collect all the reduced metal. In general the presence of any considerable amount of lead or bismuth oxides obviates the necessity of adding any collecting metal, but especial directions will be given on this point in the proper places. The R. F. must be powerful, large enough to cover the borax, and must be directed principally upon this, while the reduced metal button* lies at the side of, or partly beneath the borax, and is protected from oxidation both by the spreading flame and the glowing coal. The student must, from the

* The word *button* is always used for the *metal*, and *bead* for the *flux;* occasionally the word *globule* may be used for either, but generally with the prefix *metallic, glassy*, etc.

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first, endeavor to keep the borax bead in a spherical form, not allowing it to adhere to or spread out over the coal, as it will certainly do if any lead from the collecting button is allowed to oxidize and dissolve in it. When the bead already contains lead oxide it has a great tendency at first to spread out, but it must be brought into the spherical shape by a strong R. F. If this cannot be done, the borax glass should be cooled, cut away from the coal and turned over. It will then be easy to bring it into the spherical shape with a good R. F., and it must be kept so throughout the whole operation. This is necessary, because the collecting metal button must, from time to time, be brought into contact with every part of the melted bead, by turning the coal, so that every particle of reduced metal may be gathered into the one button. Any particles scattered through and left in the bead may afterward interfere with the tests for the non-reducible oxides. The reduction should be continued several minutes, being completed when the well-melted bead appears free from all particles of reduced metal and has been kept in quiet fusion for some moments. At no time is an O. F. to be used.

The student should practice this very important operation, using at one time two medium sized borax beads, previously rather highly charged with a mixture of about one part of copper oxide and two parts, by volume, of lead oxide, without adding any collecting metal. He should succeed in obtaining finally a spherical, clear, colorless borax bead, which, separated from the lead by gentle hammering between paper, and remelted alone on wire in O. F., should not show any green or blue color, due to copper oxide, either when hot or cold.

It is not altogether easy to remove the last traces of copper oxide from the borax when relatively much copper was originally present, even if the oil lamp is used, and the oil lamp is almost essential to success, if beads of ordinary size are to be handled; the wick, too, should be raised so as to furnish a slightly smoking flame. The student will often do well, therefore, especially when compelled to rely on the gas or other less effective flames, to again reduce the borax glass beside a bit of pure lead, since the last traces of copper can be thus more readily removed than when the lead, or other collecting metal, is already largely alloyed with copper.

The same directions, for the most part, are to be observed when treating the substance from the first on coal, as in *b*. Occasionally it is advantageous at once to wrap the powdered borax and oxides, with a collecting metal, if needed, in a soda-paper cylinder. In that case the student will find the necessary directions for performing the reduction in § 131, *b*; the O. F. is naturally never to be used and

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only such of the directions referred to are to be followed as bear on this especial operation. A careful operator can often dispense with the paper wrapper, by taking care to collect all the particles into one mass during the fusion. Gold is often preferable as a collecting metal, because it does not oxidize and dissolve in the fluxes, and therefore reduced meta's can be more readily detected in it, but lead is usually more convenient. Silver is seldom so useful, except when only the non-reducible oxides are to be regarded; it interferes especially with the detection of nickel.

The following oxides cannot be reduced from borax B.B. alone. The alkalies, earths, and oxides of iron, cobalt,* chromium, manganese, uranium, vanadium, tungsten, molybdenum, titanium, columbium, and tantalum.

Of the other metals, gold, silver, copper, and nickel, as well as more or less of the somewhat volatile lead, bismuth and tin, will be found in the reduced button; arsenic and antimony may be present alloyed with other metals, but the arsenic at least, together with tellurium, will be in most cases volatilized or remain only in traces, alloyed with other reduced metals. Zinc and cadmium are also volatilized.

When it is desirable to form a coat of any volatile metals the hole must naturally be made in a side face of the coal and the side of the hole opposite the lamp should be sloped a little, to allow the current of the blast to sweep over it.

2. General Remarks on the Salt of Phosphorus Bead.

It is made from the crystallized S. Ph.,† as described for the borax bead, 1, *a*, but requires more care, since the flux boils and easily drops from the wire. If a rather large bead is wanted the trouble can in part be avoided by making a loop with several coils, or by first fusing the salt on coal; but in general it is quite sufficient simply to melt a little of the salt at a time on an ordinary loop, until the bead is large enough, directing the flame upward a little, so as to support the flux somewhat by the blast. The flux is not to be regarded as fully melted until it is red-hot, since its first liquefaction is due simply to its water of crystallization.

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* In presence of arsenic oxide some cobalt may be reduced as arsenide, while traces of cobalt may be reduced from a borax bead very highly charged with cobalt oxide.

† Melted S. Ph. soon absorbs moisture and softens, so that it cannot be kept in the glassy state like borax glass.

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Compare the directions just given for the use of borax on coal.

3. OTHER DETAILS TO BE NOTED IN THE USE OF THE GLASS FLUXES.

Unless substances dissolve in the beads very easily they should be finely powdered, and the student must ascertain that everything has been dissolved in the melting flux, as shown by the absence of any red-hot specks floating in the bead, while still fused in O. F., because such particles will often be in a different state of oxidation from that of the fully dissolved substance, and may then at first afford, as they continue to dissolve, a different color.

Dissolve but little of a substance in the bead, when making preliminary tests at least, and add more until the desired result is attained, lest the bead should at once become so highly charged as to be opaque or give uncertain results. In general, if the object aimed at is a test of the coloring power of a substance, the student should not decide that the substance possesses no such power until a considerable quantity of the substance has been gradually added, although for special purposes it may be necessary to use only moderate quantities. The student must observe the colors imparted to the beads both when hot and cold, looking through them when held before a window, or, still better, over a well-lighted white surface, but not before the lamp flame, unless so stated. Characteristic changes of color often ensue in cooling beads. The transparency is also carefully noted under similar conditions. The lens is frequently useful in examining beads.

If for any reason it becomes necessary to remove part of a bead, fuse it in O. F., press it flat with the hammer, and then chip off the edges as much as necessary. This is often useful when too much of a substance has been dissolved and it is desirable to dilute the bead by adding more of the flux.

As already stated, § 43, sulphides (and sometimes arsenides, etc.) require roasting before they are tested with the glass fluxes.

4. TREATMENT OF METALS AND METALLIC COMPOUNDS WITH THE GLASS FLUXES, WITH PRECAUTIONS TO BE OBSERVED FOR THE PROTECTION OF THE PLATINUM SUPPORT.

Any fusible metal and many arsenides, etc., heated with platinum will alloy with and injure it. To obtain reactions directly from metals, alloys and metallic compounds with arsenic, antimony, etc., the substance is placed beside the flux, in general already melted to a glass, in a cavity on coal, which is ordinarily shallow, especially for oxidations. The O. F. or R. F., as may be directed, is then used until the desired result is produced, after which, if necessary, the

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soft glass is pinched flat with the forceps and its color examined; or it may be drawn away from the coal and remelted on wire, either in O. F. or R. F. and with addition of more flux, according to circumstances.

Platinum wire may be used as a support for the fluxes when testing in O. F. any oxide except of gold (or of silver in large quantities), but never for a metal, compound of arsenic or antimony with a metal, or a sulphide, except, in general, sulphides of the alkalies and alkaline earths. No flux containing oxides of silver, lead, bismuth, or any considerable amount of tin, copper, or nickel oxides should be exposed to the R. F. on platinum without care to avoid alloying the reduced metal with the wire, as may be the result if the R. F. is kept up too long.

FLAMING.

45. When a flux contains nearly or quite as much of a substance as it can dissolve it is said to be *saturated* or *highly* saturated, and it will remain transparent, at least if in a very thin film; but when it is *super-saturated* the undissolved part will render the bead more or less opaque, or enamel-like. A bead will often hold more of a substance in solution while hot than on cooling, and beads nearly saturated with the alkaline earths and certain other oxides will often remain clear even when cold, but will become opaque by *flaming*. To produce this result, dissolve gradually in the bead just enough of the substance to make it slightly opaque on cooling, and then add cautiously just enough of the flux to make the bead remain quite clear after being again fused and cooled. The nearly saturated, cold bead is now to be heated by an intermittent O. F., or is exposed to the O. F. and quickly withdrawn several times, so as to be alternately heated and slightly cooled, being thus kept for a short time almost, but not quite fused. After this treatment a part of the dissolved substance separates, rendering the bead *cloudy* or *opaque by flaming*, as the expression is, and the cloudiness is often due to separated crystals, which the microscope will show in the flattened bead. These crystals have been frequently studied, but without leading to any practical tests.

I. Special Tests with Borax and Salt of Phosphorus, for a Few of the Commoner Oxides.

46. *a. All metallic oxides* (except those easily reduced to metal by heat alone, such as the oxides of gold and the metals of the platinum

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group), dissolve in borax in O. F. more or less readily, unless too easily vaporizable.

All the substances enumerated in the Tables II. to V., § 48, are soluble in S. Ph. also, but silica is so slightly soluble, even in fine powder, that it can be easily detected by this means.

It will be seen that S. Ph. often gives different colors from borax with the same oxide; it also frequently gives similar colors, but not so deep.

In this paragraph will be given the most characteristic reactions of certain oxides, for practice with the glass fluxes and to acquaint the student with the most frequently needed tests. They should be carefully studied, and the platinum wire is to be used, unless otherwise directed. Care must be taken to use a pure O. F., § 26, and R. F., § 27, and especially with substances like oxide of chromium, see *j*, involving sometimes slight differences in the colors of the glass beads, or else correct results may not always follow. The same bead that has been used for O. F. will generally serve for R. F., but borax and S. Ph. are never mixed. A glass bead which is to be treated with tin on coal, as in *c*, *f*, *h*, etc., must be separated first from the wire, best by shaking it off, § 44, 1, *c*. If the result described is not obtained, the test should be repeated until it is.

For a general view of the behavior of all the oxides with the glass fluxes see Tables II. to V., § 48.

b. Silica [54]. In S. Ph. scarcely dissolves at all, unless in very fine powder, and even then the greater part forms a semi-transparent undissolved residue, or remains in minute particles, best seen by aid of the lens. In case of silicates whose bases dissolve easily in S. Ph. a very small splinter of the mineral should be heated with the S. Ph. in O. F., when the bases will dissolve, perhaps coloring the bead, and will usually leave a silica skeleton behind [44]. See also § 112, *a*.

c. Antimonous oxide [30]. A small quantity is dissolved in S. Ph. in O. F., the colorless bead shaken off, § 44, 1, placed beside a bit of tin smaller than the bead, in a moderately deep cavity in charcoal, and heated with the R. F. *only long enough* to *fuse the two together*, when the S. Ph. bead will become gray, brown, or black, from the presence of reduced, metallic antimony. A deposit of soot on the bead must be avoided, and the student must not try to dissolve the tin in the S. Ph., but only to make the two adhere together. See also § 96, *b*, and compare remarks under tin as a reagent, § 12, 11.

d. Bismuth oxide [36]. Try the same test as with antimonous

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oxide, above. Less bismuth is needed to produce an equally dark bead. See § 90, *a*. The dark appearance is due to metallic bismuth.

e. Tungstic oxide (also called *tungstic acid*) [55]. The slightly charged borax bead is nearly or quite colorless when hot, colorless when cold. The moderately saturated bead is yellowish while hot, colorless on cooling; in R. F. is not changed unless somewhat highly charged, when it becomes yellowish while hot, yellowish-brown on cooling. In S. Ph. in O. F. the somewhat highly charged bead is yellow while hot, colorless on cooling. In R. F. this glass becomes dirty green while hot, and blue (very characteristic) on cooling, unless treated too long, when it is bluish-green. If iron is also present (add a little of [8]) the S. Ph. bead in R. F. is yellow while hot, brownish-red to blood-red on cooling, but it can still be made blue by treatment beside tin on coal in R. F., unless too much iron is present.

f. Titanic oxide (or *acid*) [56]. (Rutile contains more or less iron oxide and will ordinarily show it by giving a distinctly yellow bead while hot, with borax, while the S. Ph. bead may not show pure titanium reactions, but may indicate the presence of the iron also. By careful reduction, however, the S. Ph. bead will finally become violet when cold.)

In borax behaves much like tungstic oxide; a very highly charged bead may be flamed enamel-blue in R. F., but it is not necessary to try this test. In S. Ph. in O. F. the somewhat highly saturated bead is yellow while hot, colorless on cooling. In R. F. this bead is still yellow while hot, but reddens in cooling, and at last shows a very characteristic violet color (titanous oxide).

In presence of iron (add a little of [8]) the S. Ph. bead in R. F. behaves like the similar tungstic oxide bead containing iron, *e*, but with tin on coal becomes violet, unless too much iron is present.

g. Molybdic oxide (or *acid*) [1]. The moderately charged borax bead in O. F. is yellow while hot, colorless on cooling. In R. F. the bead, previously rather highly charged in O. F., becomes brown or nearly black and opaque, according to the saturation, from formation of the lower binoxide of molybdenum. After a strong R. F. this may be seen as separate black flakes in the flattened glass, which itself will be yellowish-green. In S. Ph. in O. F. a moderate amount gives a yellowish-green glass while hot, becoming nearly colorless when cold. In R. F. this same glass becomes dirty green while hot, pure green on cooling.

h. Iron sesquioxide (or *ferric oxide*) [57]. With borax in O. F.

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the slightly charged bead is yellow or yellowish while hot, colorless on cooling. The moderately saturated bead is reddish when hot, becoming yellow on cooling, while a still more saturated bead is dark red when hot, dark yellow on cooling. In the absence of other and rarer oxides, as of uranium, vanadium and cerium, which also color the cold glass yellow when present in moderate quantity, the borax bead in O. F. alone furnishes an excellent test for iron ; chromium gives a greenish-yellow to yellowish-green glass.

In R. F. the moderately saturated glass becomes bottle-green (brownish-green or dark yellowish-green, like cheap bottle glass ;) on coal beside tin in R. F., the bead becomes clear vitriol-green (clear, pale green), after long enough blowing. The bottle-green color is due to proto-sesquioxide of iron ; the vitriol-green to simple protoxide of iron or ferrous oxide. The presence of a considerable excess of oxide of iron renders the borax bead nearly black in R. F.

With S. Ph. in O. F. a moderate amount gives a yellowish red glass while hot, becoming yellow, then greenish, and finally colorless on cooling. Very much oxide of iron gives a dark red glass while hot, becoming brownish-red, then dirty green, and finally yellow. In R. F. a little oxide gives no color, but more gives a red glass while hot, becoming yellow, greenish, and finally smoky brown, with a slight reddish tinge if highly saturated. Oxides of iron color S. Ph. less intensely than they do borax.

i. Uranic oxide [58]. In borax in O. F. the colors are much as with iron sesquioxide, but somewhat lighter for equal amounts, while a highly saturated glass can be flamed to a yellow enamel. In R. F. like iron oxide, but with tin on coal becomes dark green (uranous oxide).

In S. Ph. in O. F. a glass, yellow while hot, yellowish-green when cold. In R. F. this glass is dirty-green while hot, fine pure green (uranoso-uranic oxide) on cooling, and somewhat deeper green with tin on coal (uranous oxide).

j. Chromic oxide [59]. The slightly charged borax bead in O. F. is yellow while hot, pale greenish-yellow when cold ; the moderately charged bead is dark yellow to dark red when hot, becoming lighter yellow in cooling, and finally yellowish-green. In R. F. the bead is fine green when cold. The S. Ph. bead is green when cold after treatment with either flame.

A very pure O. F. is necessary to obtain the yellowish-green color with borax ; a very little reducing action turns the bead green.

k. Cobalt oxide [60]. A blue glass with either flux and in either flame. The oxide colors so strongly that too much may easily be

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used and the bead will appear black, unless flattened or drawn out in a thread.

l. Copper oxide [3]. With borax in O. F. a little oxide gives a bead that is green while hot, and blue when cold; a larger quantity gives a dark green or opaque glass while hot, greenish blue when cold.

In R. F. the highly charged glass when cold will be opaque red, from presence of suboxide of copper; it should not be treated too long, lest the reduced copper alloy with the wire. If the bead is treated with R. F. on coal* the copper is finally reduced to metal, while the glass, pinched or drawn out, will be colorless. With tin on coal in R. F. a somewhat highly charged bead assumes a brownish-red to opaque red color on cooling.

With S. Ph. the reactions are much the same as with borax in both flames and on coal, but the S. Ph. bead with tin affords a more delicate test for copper than the borax bead. See § 88, *d.*

m. Nickel oxide [61]. In O. F. the slightly charged borax bead is violet while hot, pale brownish on cooling; the colors of the moderately saturated bead are similar but deeper, and the cold bead is reddish-brown. This change of color in cooling is very marked. In R. F. the nickel is reduced to metal, rendering the glass grayish, or opaque if much is present; a longer blast collects the metal together, leaving the glass itself colorless. This result is more readily obtained with tin on coal, the reduced nickel alloying with the tin.

In S. Ph. in O. F. a reddish glass, becoming yellow to reddish-yellow when cold (yellowish-red if nearly saturated). In R. F. on wire this bead is unaltered, but with tin on coal behaves like the borax glass.

n. Manganese sesquioxide. (Use the binoxide [2].) In borax in O. F. the very slightly charged bead is violet (amethystine) while hot, violet-red on cooling. A slight excess renders the bead very dark, so that the red color can only be seen after it is flattened to quite a thin film.

In R. F. a moderately dark glass is readily made colorless, but if very dark it can scarcely be made so, except beside tin on coal. The bead in R. F. becomes colorless because the manganese is reduced to the very feebly coloring protoxide.

With S. Ph. in O. F. the glass is brownish-red while hot; reddish-violet when cold. In R. F. the glass is more readily made colorless than with borax.

* All such reductions on coal should be made in a moderately deep cavity; see § 7.

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J. Tables Showing the Characteristic Behavior of Oxides with the Glass Fluxes.

47. The following Table I. is designed for practice on the oxides most frequently tested by the glass fluxes alone. In using it the student should first employ slightly charged borax beads and afterward add more of the substance to be tested. The S. Ph. beads can be moderately charged at first and afterward more highly, if necessary to produce a decisive result. (When rutile is given as an example of the behavior of titanic oxide, as is often the case, it should be remembered that it contains more or less oxide of iron, which will cause it to color both the borax and S. Ph. beads a little deeper yellow than would pure titanic oxide.)

When the student is in doubt about the depth of the yellow color produced by the oxides of division 1 and 3 of the table, he can always decide which substance he has in hand by following out the subsequent tests, especially those in R. F.

TABLE I.—Behavior of a few of the Commoner Oxides with Borax and Salt of Phosphorus.

Borax in O. F., hot.	Borax in R. F., cold.	S. Ph. in O. F., cold.	S. Ph. in R.F., cold.
1. Distinctly yellow if only slightly charged, but perhaps colorless on cooling. If highly charged, red to deep yellow; paler yellow on cooling.	If moderately charged bottle-green.	Yellow to colorless.	Smoky reddish-brown. *Iron.*
		Greenish-yellow to colorless.	Green. *Uranium.*
2. (Cold). If rather highly charged greenish-yellow to distinctly green, with slight yellowish tinge.	Fine green, even if quite moderately charged.	Yellow if somewhat highly charged.	Green. *Vanadium.*
		Green.	Green. *Chromium.*
3. If somewhat highly charged slightly yellow, but colorless on cooling.	Yellowish to dark brown, if somewhat highly charged.	Colorless or nearly so.	Blue. *Tungsten.* Violet. *Titanium.*
	Brown to black, if somewhat highly charged.	Colorless or nearly so.	Green. *Molybdenum.*
4. Violet; reddish brown on cooling.	Gray to colorless.	Yellow; red while hot if at all highly charged.	Yellow. *Nickel.*
5. Amethyst-red; violet-red on cooling.	Colorless.	Reddish-violet.	Colorless. *Manganese.*
6. Blue; same when cold.	Blue.	Blue.	Blue. *Cobalt.*
7. Green; greenish-blue to blue on cooling.	Colorless or reddish.	Greenish-blue or blue.	Colorless or reddish. *Copper.*

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TABLES SHOWING THE CHARACTERISTIC BEHAVIOR OF OXIDES IN GENERAL WITH THE GLASS FLUXES.

48. The following four Tables, altered slightly from Plattner, 5th ed., Leipsic, 1878, contain a very comprehensive view of the behavior of all the oxides which give more or less characteristic reactions with borax and salt of phosphorus.

TABLE II.—The Borax Bead in O. F. is:

1. *Colorless.*

Hot and cold:	Silica, alumina, binoxide of tin.	
	Baryta, strontia, lime, magnesia, glucina, yttria, zirconia, thoria; oxides of lanthanum and silver; tantalic and tellurous oxides.	Cloudy (white) on cooling if highly saturated.
	Titanic, tungstic and vanadic oxides; molybdic trioxide, oxides of didymium, indium, zinc, cadmium, lead, bismuth and antimony.	If only slightly saturated.

2. *Yellow.*

Hot:	Titanic, tungstic, columbic and molybdic oxides; oxides of zinc, cadmium and lead (all seven opaque on flaming).	When highly saturated. Colorless on cooling.
	Oxides of bismuth and antimony.	
	Oxides of cerium, uranium, and iron (all pale to colorless on cooling).	In small quantity.
	Oxide of chromium; greenish-yellow on cooling.	
	Vanadic oxide (moderately saturated); greenish-yellow on cooling.	

3. *Red to brown.*

Hot:	Oxides of iron, cerium and uranium; all yellow on cooling, and the last two enamel-like by flaming.	In moderate to large quantity.
	Chromic oxide, yellowish-green on cooling.	
	Oxide of iron with manganese, yellowish-red on cooling.	
Cold:	Oxide of nickel, reddish-brown to brown.	Violet while hot.
	Oxide of nickel with little cobalt, violet-brown.	
	Sesquioxide of manganese, violet-red.	

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4. *Violet (or amethyst-red).*

Hot:
- Sesquioxide of manganese, violet-red when cold.
- Oxide of nickel, reddish-brown to brown when cold.
- Oxide of nickel with little cobalt, violet-brown when cold.
- Oxide of nickel with considerable cobalt. } Also cold.
- Oxide of cobalt with manganese. }

5. *Blue.*

Hot: Oxide of cobalt ; the same on cooling.

Cold: Oxide of copper (greenish-blue with much copper); green while hot.

6. *Green.*

Hot:
- Oxide of copper, blue to greenish-blue on cooling.
- Oxide of iron with cobalt or copper ; oxide of copper with iron or nickel. } In cooling becomes light green, blue or yellow, according to the proportion of the different oxides present and the degree of saturation.

Cold:
- Chromic oxide; moderately saturated (yellowish-green); yellow to red while hot.

The bead previously examined in O. F. is now treated in R. F., observing the directions in § 44 *et seq.*

TABLE III.—The Borax Bead in R. F. is:

1. *Colorless.*

Hot and cold:
- Silica, alumina, oxides of tin, indium and manganese; the last tends to become slightly red on cooling.
- Baryta, strontia, lime, magnesia, glucina, yttria, thoria, zirconia; oxides of lanthanum and cerium; tantalic oxide. } Opaque by flaming if highly saturated.
- Columbic oxide, in small quantity.
- Oxides of silver, zinc, cadmium, lead, bismuth, antimony, nickel; tellurous oxide. } By long blowing ; gray after brief treatment.
- Oxide of copper; if highly saturated, opaque red on cooling.

2. *Yellow and brown.*

Hot:
- Titanic oxide, yellow to brown; if highly saturated, enamel-blue on flaming.
- Tungstic oxide, brownish on cooling.
- Molybdic trioxide, brown to opaque and nearly black. } If highly saturated.
- Vanadic oxide, brownish; emerald-green on cooling.

3. *Blue.*

Hot: Oxide of cobalt; also when cold.

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4. *Green.*

Hot and cold:
- Oxide of iron, yellowish to dark bottle-green, especially when cold; nearly black if containing a large excess.
- Oxide of uranium, yellowish-green; black by flaming if highly saturated.
- Oxide of chromium, light to dark emerald-green, according to its saturation.

Cold:
- Vanadic oxide, emerald-green; brownish while hot.

5. *Gray or cloudy.*

(Often becoming so during the blast.)

Cold:
- Oxides of silver, zinc, cadmium, lead, bismuth, antimony, thallium, nickel, tellurous oxide. — After short treatment; by longer blowing, colorless.
- Columbic oxide, highly saturated.

6. *Red.*

Cold:
- Oxide of didymium; rose colored, if very highly saturated.
- Oxide of copper, if highly saturated and not well reduced, opaque red; sometimes clear ruby-red.
- Oxide of manganese, only if highly saturated and not well reduced; the color is faint and sometimes only in streaks.

TABLE IV.—The Salt of Phosphorus Bead in O. F. is:

1. *Colorless.*

Hot and cold:
- Silica, very slightly soluble.
- Alumina, oxide of tin; slowly and with difficulty dissolved.
- Baryta, strontia, lime, magnesia, glucina, yttria, thoria, zirconia; oxides of lanthanum and didymium; tellurous oxide. — Cloudy (white) by flaming.
- Tantalic, columbic, titanic and tungstic oxides; oxides of zinc, cadmium, indium, thallium, lead, bismuth, and antimony. — If not too highly saturated. Yellowish to yellow while hot, if nearly saturated; colorless on cooling.

2. *Yellow.*

Hot:
- Titanic, columbic, tantalic and tungstic oxides; oxides of zinc (enamel-like by flaming), cadmium, lead, bismuth and antimony. — If nearly saturated; colorless on cooling.
- Oxide of silver, yellowish; opalescent when cold.
- Oxides of iron and cerium; if slightly saturated, colorless on cooling; if highly saturated, red while hot, yellow when cold.
- Oxide of uranium, yellowish green on cooling.
- Vanadic oxide, dark yellow; paler when cold.

Cold:
- Oxide of nickel, reddish while hot.
- Oxides of iron and cerium, highly saturated.

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3. *Red.*

Hot:
- Oxides of iron and cerium, highly saturated ; yellow when cold.
- Oxide of nickel, reddish ; yellow when cold.
- Oxide of chromium, reddish ; emerald-green when cold.

Cold: Oxide of manganese, reddish-violet; brownish-violet while hot.

4. *Blue.*

Hot: Oxide of cobalt, smalt-blue ; also when cold.

Cold: Oxide of copper, sky-blue ; green while hot, greenish-blue on cooling, if highly saturated.

5. *Green.*

Hot:
- Oxide of copper, blue on cooling, or greenish-blue if highly saturated.
- Molybdic trioxide, yellowish-green; lighter when cold.
- Oxide of iron with copper or cobalt; oxide of copper with iron or nickel. — Light green to blue or yellow on cooling, according to the varying proportions of the oxides and the degree of saturation.

Cold:
- Oxide of uranium, yellowish-green; yellow while hot.
- Oxide of chromium, emerald-green; reddish while hot.

TABLE V.—The Salt of Phosphorus Bead in R. F. is:

1. *Colorless.*

Hot and cold :
- Silica, very slightly soluble.
- Alumina, oxide of tin ; slowly and with difficulty dissolved.
- Baryta, strontia, lime, magnesia, glucina, yttria, zirconia, thoria, oxide of lanthanum. — Cloudy (white) by flaming, if highly saturated.
- Oxides of cerium, didymium (unless highly saturated) and manganese.
- Tantalic and tellurous oxides; oxides of silver, zinc, cadmium, indium, lead, bismuth and antimony. — Gray after short, colorless after longer treatment.

Cold : Oxide of iron, in moderate quantity.

2. *Yellow, Brown, or Red.*

Hot :
- Oxide of iron, yellow to red ; on cooling, first greenish, then smoky reddish-brown.
- Titanic oxide, yellow ; violet when cold.
- Columbic oxide, yellow ; brownish if highly saturated.
- Vanadic oxide, brownish ; emerald green when cold.
- Titanic or tungstic oxide with iron, yellow ; brownish-red to blood-red when cold.
- Columbic oxide with iron, brownish-red ; dark yellow when cold.

Cold : Oxide of copper, opaque red ; if highly saturated.

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3. *Violet or Blue.*

Cold:
- Oxide of cobalt, blue; also while hot.
- Tungstic oxide, blue.
- Titanic oxide, violet; yellow while hot.
- Oxide of didymium, violet: if highly saturated and after long treatment.

4. *Green.*

Hot:
- Oxide of copper; if highly saturated dark green; opaque red on cooling.

Cold:
- Oxide of uranium.
- Oxide of chromium; reddish while hot.
- Vanadic oxide; brownish while hot.
- Molybdic trioxide; dirty green while hot.

5. *Gray to Black and Cloudy.*

(Often becoming so during the blast.)

- Oxides of silver, zinc, cadmium, indium, nickel, thallium, lead, antimony, bismuth (the last two dark gray to brown or black, even in small quantity, if on coal beside tin); tellurous oxide. — Best on coal; colorless after longer treatment.

6. *Red.*

Cold:
- Oxide of copper, if highly saturated; opaque red.

K. Examination with Soda.

49. This flux, which is really *carbonate of soda* (see § 12, 1), greatly facilitates the reduction of many metallic oxides to metal, and is very useful in the decomposition of salts, combining with the acid, so that the metal is readily reduced. It is thus used for decomposing sulphides, chlorides, sulphates, phosphates, etc. Soda is also much used to decompose salts insoluble in acids. Thus certain silicates are fused with it so as to form silicate of soda, while the bases are set free and can be dissolved in acids.

Reduction of oxides and salts with soda on coal.

50. *a.* The substance should generally be in fine powder, and is to be mixed to a stiff paste with about three times its volume of soda and a little water, after which it is treated in a moderately deep cavity on coal with a strong R. F. for two or three minutes, a little more soda being added to the cooled and powdered assay if the mixture appears to be very infusible, as is often the case with tin oxide,

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for example. Whenever the formation of a coat is a principal object of the reduction, as with zinc minerals, the cavity should be shallower and the side of it farthest from the lamp should slope gently, so that the coat may form well on the coal, § 34. Sometimes the substance, especially if easily decomposed or fused, may be heated in small fragments covered by the soda, nor is it always necessary to moisten the soda. Sulphides of copper, silver, lead, bismuth, antimony, cadimum, zinc, and tin, as well as some other sulphides, yield characteristic reactions when treated raw, but in general sulphides should be roasted before treatment with soda.

If any considerable quantity of an easily fusible metal, lead, copper, etc., is present, it will collect in one or more buttons and may be easily separated from the slag of fused soda, part of which will have sunk into the coal. The buttons are detached with the knife point or picked out from the slag and adhering coal, after this has been crushed on the anvil, between paper if very small globules only are expected. The beginner will always do well not to omit the washing, § 51, if he does not find globules large enough to handle without it, and in any case washing should be resorted to for the detection of small quantities of reduced metals.

The addition of a little borax to the soda is often useful, especially with rather infusible bodies like oxide of tin, but it is not always admissible. It would, for instance, prevent the reduction of iron and cobalt. On the other hand, it is often required, as when oxides of copper or tin are to be reduced from mixtures with iron salts or oxides, when the metal is to be obtained free from reduced iron. Its use will be indicated in the proper places.

b. The reduction, unless there is reason to fear that traces of rather volatile metals, lead, bismuth, etc., will be lost, should be continued until a considerable part of the soda, if used without borax, has sunk into the coal, when the globules of metal often appear on the surface of the coal, as shining buttons. In case of non-volatile metals, like silver, gold, or copper, it is well to heat the reduced metal for some time in a rather oxidizing flame, and if it does not then show its true color on being hammered out it is again melted, and if it does not yet show the true color of one of the metals (or such as would result from their alloys with each other), but still presents a black or dark surface, due to some sulphur remaining with it, it is again treated in O. F., beside borax, to brighten its surface and remove the last portions of sulphur. Students very often fail to thoroughly reduce such compounds, and are misled by the appearance or brittleness of the resulting metallic buttons, when the

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difficulty really lies in too short a treatment with the soda or in the presence of slight impurities that can be removed as above.

When the substance is hard to reduce, like tin oxide, the addition of fine charcoal dust, in quantity about equal to the volume of the substance, will be found very useful. It almost obviates the necessity of using potassium cyanide or oxalate, although these are more powerful reducing agents than soda alone. Potassium cyanide is especially useful in the reduction of tin chlorides.

The properties of the metals thus reduced are to be examined as in § 51.

51. When substances are reducible with difficulty the R. F. should be continued until most of the soda, of which successive additions may be needed, has sunk into the coal. The fused mass and the coal just around and beneath it are then cut away and gently crushed with a few drops of water, to break up the soda slag and coal, without rubbing hard enough to grind the reduced metals to fine powder or flatten them to too thin scales, which might be washed away. The mortar is then filled with water, which is stirred, allowed to settle a moment and poured off, with the coal powder and dissolved soda. The washing, which is often necessary even with easily reducible metals, if present in small quantity, is repeated, with further crushing and gentle rubbing of the residue, until any reduced metals are obtained in a clean state, as buttons, scales, or powder, which remain in the mortar. The mass must sometimes be finally rubbed to quite fine slime, in order to wash away all coal and slag and detect minute quantities of reduced metal, but this is to be carefully done.

If necessary, the residue is examined with the magnifying-glass. Metallic, shining streaks, left on the mortar where the pestle has rubbed it, will often reveal traces of metal otherwise scarcely perceptible. (The mortar is cleansed by means of nitric or hydrochloric acid, or simply by rubbing with powdered bone-ash or very finely powdered quartz rubbed on it with the fingers.)

The reduced metals are either picked out with the forceps, or, if too small for this, are gathered into the last drop of water in the mortar and transferred to the coal with this drop, which is dried off by a very gentle flame. Magnetic residues can be gathered with the magnet.

The metals most frequently tested for in this reduction with soda can be identified as follows. (Compare also each reference, and consult § 52 for a full statement of the results of the soda reduction.) *Copper*, § 87, and *gold*, § 92, *a*, by their colors; *silver*, § 83, retains

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its bright, white lustre after being fused in a gentle O. F. and scarcely yields any coat; *tin*, § 94, *a*, also a white metal, becomes quickly covered with a dull, white coat of oxide when fused in a gentle O. F. and easily yields a white coat when rather highly heated (*antimony*, § 95, is occasionally obtained in buttons or powder); *lead*, § 85, and *bismuth*, § 89, appear as whitish to light grayish metals in small buttons, although lead has in fact a bluish lead-gray color, and bismuth is white with a slight reddish tinge. They are distinguished from each other as in §§ 86 and 90, *b*. *Iron*, § 73, *cobalt*, § 75, and *nickel*, § 77, are occasionally obtained, either as blackish-gray to gray powders, or in steel-gray to whitish, shining scales, and are magnetic; a fact best ascertained by testing the powder under water in the mortar with the magnet. After ignition in O. F. on coal, they will yield characteristic colors with borax and S. Ph.

When there is too little metal present to handle in this way (although very small quantities can be so treated), the metal can be wiped from the mortar with a small piece of filter paper, which is rolled up, dried and burned in a very gentle O. F. on coal, the ashes being further treated on coal to get coats, or with appropriate fluxes. When the latter are used they can be at once rolled up with the paper and metal and treated on coal until the paper is burned, leaving the flux with the oxidized metal, dissolved in it, to be treated afterward on wire if desirable. The student should try a reduction test on tin oxide [33] with soda alone, when he may wash the metal in the mortar, and also with soda and charcoal dust. Even with the gas flame a reasonably good reduction of tin oxide with soda alone will result in globules large enough to be handled with the forceps, although, if coal dust is not used, they may not be readily detected without washing in the mortar.

52. The reduction test with soda is of great use for detecting traces of tin in presence of non-reducible metals, and also for determining minerals and recognizing certain reducible metals.

Iron, nickel, cobalt (all yielding magnetic metals), tin, indium, bismuth, lead, tellurium, antimony (all yielding more or less of their characteristic coats at the same time, although tin may fail to yield a very perceptible coat), and copper, as well as the noble metals, gold, silver, platinum, etc., can all be obtained as metals by reduction with soda (bismuth, lead, copper, and the noble metals, very easily), and partly or entirely recognized by this test alone. Antimony, tellurium, and indium are reduced easily, but are very readily volatilized, and hence not so certainly obtained in the metallic state, unless present

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in somewhat considerable quantity. Traces of lead and bismuth may also be lost by too long treatment in R. F.

The very volatile metals, arsenic and mercury, do not remain with the soda, but pass off in vapor; arsenic usually yielding a copious, very volatile, white coat of arsenous oxide, and mercury yielding no coat, or but a very slight, grayish film of metallic mercury, far from the assay and very volatile. Traces of metallic cadmium can be obtained by washing the soda, if much cadmium oxide is present, and the reduction made at a moderate temperature; while traces of zinc may remain alloyed with any copper also reduced; but the student may regard both cadmium and zinc as practically not to be obtained in the free state by this test, since they pass off in vapor, yielding coats of their oxides. Tungsten and molybdenum are also obtained as metals, in the form of infusible powders.

Alloys are usually formed when more than one metal is present, arsenic yielding a brittle alloy with cobalt, iron, nickel or copper, and antimony and tin forming brittle alloys with copper especially. Iron and copper do not readily alloy together, and the addition of a little borax will often be advantageous in reducing compounds containing iron, since it keeps the iron dissolved as oxide and aids thus especially in certain tests for tin, copper, and zinc.

FORMATION OF COATS WITH SODA ON COAL.

53. The operation is conducted as above, but in a shallow cavity if the substance is not at the same time to be tested for non-volatile metals, difficult to reduce. In this latter case the cavity should be moderately deep, but shaved off to a rather gentle slope on the side farthest from the lamp, so as to allow any coat to be properly deposited on the coal.

The R. F. is continued long enough to ensure the volatilization of any metals that form coats, characteristic coats of oxides being in this way readily obtained from compounds of zinc, cadmium, indium, arsenic, antimony, tellurium and lead, which might otherwise be volatilized as chlorides, etc., in coats not so characteristic, or might fail to be volatilized at all. (When the colored flame afforded by a coat is to be used as a test it is better to avoid using soda, if possible.)

The soda is especially useful in obtaining a coat of lead oxide from the phosphate, selenide and certain sulphides containing also antimony, as well as for reducing the volatile metals generally from their silicates, chlorides, bromides and iodides. The addition of borax is

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often useful in testing for zinc when much iron is present, or when the substance is not easily reduced by soda alone, as in gahnite (zinc spinel) and in franklinite (oxides of iron, manganese and zinc).

Small quantities of antimony with copper, or of arsenic with certain metals, especially nickel and cobalt, may not yield any coats by this treatment alone; see §§ 96, *d*, and 97, *a*.

Note.—For reduction with soda in closed tube, see the directions given under mercury, § 84, *a*.

FUSIBILITY OF SUBSTANCES WITH SODA.

54. The substance is mixed in fine powder with soda and a little water to a stiff paste, and is then spread in a shallow cavity on coal, gently dried and finally strongly heated in O. F. The test is mainly useful in distinguishing minerals, and is not often employed. The most important results that may be obtained are as follows:—

a. Effervescence of the fusing mixture, caused by an acid. Silicic, boric, and titanic acids yield, with *just enough* soda, clear beads; those with silicic or boric acid remaining also clear when cold. Tungstic and molybdic acids combine with the soda also, but sink into the coal.

b. Absorption of the fused mass by the coal. Salts of barium [51] and strontium, like the sulphates and carbonates, fuse with the soda and sink into the coal, while those of calcium (lime) [27], magnesium, and aluminium, although decomposed, do not sink in with the soda, but the bases remain as an infusible crust or mass upon the coal, after the soda salts have been absorbed.

Silicates of the above bases will leave a residue of sodium silicate, forming a more or less fusible bead or scoria with the unabsorbed bases. If a substance is not attacked at all by the soda it will be left behind unaltered when the soda has been absorbed. In all of these tests it is desirable to add the soda by degrees, since the fusibility of many salts varies greatly in proportion to the amount of soda used.

For other tests with soda see manganese, § 72, *c*, and sulphur, § 103, *c*.

L. Examination with Cobalt Solution.

55. For this test always use the O. F., remembering in certain cases where the directions seem to contradict this rule, that a substance heated by the outer part of the R. F. is exposed to the oxidizing action of hot air.

Fragments of minerals, unless too compact to absorb the cobalt

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solution, may be tested in the platinum forceps; powders sometimes on platinum wire. In general, however, the substances can be satisfactorily tested in a very shallow cavity on coal, being moistened with a drop of the cobalt solution and gently dried in O. F., after which they are heated by a strong O. F. Powders should be spread thinly on the coal, after being moistened with the solution, and compact minerals, unless they can be made porous by previous ignition, should be finely powdered before the test.

Salts which swell up or liquefy in their water of crystallization, such as magnesium sulphate, alum, and many salts soluble in water, should first be ignited alone until they leave an infusible crust. Coats on coal are simply moistened *when cold* with a drop of the solution. The cooling is hastened by placing the anvil on the coat. The resulting colors are brighter and purer in proportion to the duration and intensity of the ignition, and appear in their true tints only after the substance has cooled. No regard is to be paid to the purplish or red color, which may be caused simply by the drying of the pink cobalt solution, before ignition. Too much strong cobalt solution may obscure the true result by forming an excess of black oxide of cobalt, and therefore only moderately strong solution is used, adding successive portions if necessary, until experience has been obtained.

In general no decisive results will be obtained from substances which in O. F. remain dark after strong ignition, before any cobalt solution has been used. Fusible salts frequently give a blue glass, the color of which is due to the cobalt alone. This is especially to be remembered when testing for alumina, § 66, and the student must be very careful not to add any kind of flux. Should the coal ash alone give a distinct color (G. J. Brush) with cobalt solution, the coal must be used with judgment.

The following are used as characteristic reactions:

Flesh-color or pink (only after intense ignition and often *pale brownish-red* if a little iron is present). Magnesia [62]. Magnesite, finely powdered and many magnesium minerals give but a feeble tint; the artificial and more porous carbonate shows it more strongly.

Violet. Magnesium arsenate, borate or phosphate (fusible).

Blue. Alumina [63]. Ignite the alum first alone to full redness.

Green. Zinc oxide. Form some as a coating on coal from zinc [31] (yellowish-green or grass-green). Tin binoxide. Form an abundant coating from tin [32] (bluish-green).

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Practice alone will acquaint the student with these, and especially with the difference between zinc and tin; compare §§79 *b*, and 94 *b*. Other substances yield distinct colors with cobalt solution, but they are not used as characteristic tests, and are given here to forewarn the student against mistakes. They are:—

Brownish-red, baryta; *flesh color*, tantalic acid; *violet*, zirconia; *blue*, silica (pale reddish to purplish-blue); fusible salts (smalt-blue, often glassy and perhaps modified by other colors); *green*, antimonic acid (dirty, dark green); titanic acid (yellowish green); columbic acid (dirty green); *gray*, strontia, lime, glucina (bluish-gray).

The green color due to antimonic acid is apt to deceive the beginner into believing that a zinc or tin coat is present, unless proper care is taken; see *Notes*, §§ 79, *d*, and 94, *e*; silica and fusible salts may possibly be mistaken for alumina, but apart from these the student will not be likely to be misled by any of the above bodies. The blue yielded by alumina is *always dull* and *unfused* (in case of silicates of alumina it may show a very feeble lustre), and hence any decidedly glassy or fused blue compounds are not to be regarded as indicating alumina with certainty. If fusible salts of alumina are present, as alum, they should first be heated until they leave an infusible residue, and this should be tested with cobalt solution. Too high a temperature should also be avoided in doubtful cases.

The lens must be used to examine coats, powders, and even the apparently uncolored coal itself, after the ignition with cobalt solution, since minute specks of the desired color may often be thus detected, sometimes almost hidden beneath fibres of the coal or under the heated mass, while any traces of fusion can also be recognized.

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TABLE VI.

BEHAVIOR OF THE

ALKALIES, EARTHS, AND METALLIC OXIDES, ALONE AND WITH REAGENTS, BEFORE THE BLOWPIPE.

(Translated from Plattner.)

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Table VI., A. ALKALIES.	ALONE ON PLATINUM WIRE.	ALONE ON PLATINUM FOIL.	REMARKS.
1. POTASSA, K̇.*	Colors the flame violet; a very little soda prevents the reaction.	0	In solution, color red litmus paper blue.
2. SODA, Ṅa.	Colors the flame strongly reddish-yellow, even when very much potassa is present.	0	
3. LITHIA, L̇i.	Colors the flame carmine-red, even when more potassa than lithia is present; but a little soda alters the flame to yellowish-red or reddish-yellow	When fused turns the foil about it dark yellow; on washing and igniting the foil this disappears, but the foil loses its polish and appears dull, especially when heated to redness.	
4. AMMONIA, N H³.	In combination with nitric and sulphuric acids and chlorine gives a pale greenish flame.	0	Recognized by its odor; also colors red litmus paper blue.

Table VI., B. EARTHS.	ALONE ON CHARCOAL AND IN THE FORCEPS.	WITH BORAX ON PLATINUM WIRE.	WITH SALT OF PHOSPHORUS ON PLATINUM WIRE.	WITH SODA ON CHARCOAL.	WITH COBALT SOLUTION IN THE O. F.
1. BARYTA, Ḃa.	The hydrate fuses, boils, swells, becomes fixed on the surface, and then sinks into the coal with violent ebullition. The carbonate fuses to a clear glass easily, and becomes enamel-white on cooling. On repeated fusion it becomes caustic and is absorbed. Colors the flame yellowish-green.	The carbonate dissolves to a clear glass with effervescence, and can be flamed enamel-white with a certain amount; with more it becomes enamel-white of itself on cooling.	As with borax.	Fuses with it and is absorbed by the charcoal.	Fusible to a brownish-red bead, which loses color on cooling, and soon crumbles to a light-brown powder in the air. With very dilute solution the bead only appears pale brown.

* The oxides of *rubidium*, Ṙb, and *caesium* C̈s, show quite similar reactions.

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2. STRONTIA, Ṡr.	The hydrate behaves like baryta. The carbonate fuses only on the edges, ramifying like cauliflower; the projections are luminous, tinge the R. F. red, and have an alkaline reaction. In the forceps, colors the flame crimson.	Like baryta.	Like baryta.	Ṡr is insoluble. Ṡr C̈ fuses with an equal amount of soda to a clear glass, milk-white on cooling. More strongly heated the glass boils and the caustic earth is absorbed. If more is added it does not dissolve, but becomes caustic and goes into the coal.	Sinters and assumes a black or dark gray color.
3. LIME, Ċa.	Ċa is unaltered. Ċa C̈ becomes caustic, whiter, more luminous, then alkaline, and crumbles to powder if moistened. In the forceps, colors the flame feebly red.	Dissolves easily. The clear glass can be flamed opaque. Ċa C̈ dissolves with effervescence. With more the glass becomes cloudy and crystalline on cooling, but never so milk-white as with baryta or strontia.	Dissolves largely (with effervescence, carbonate) to a clear glass, that can be flamed opaque, when rather saturated. Perfectly saturated the glass becomes milk-white on cooling.	Insoluble. The soda goes into the coal, leaving lime behind.	Perfectly infusible; becomes gray.
4. MAGNESIA, Ṁg.	The carbonate is decomposed, becoming luminous and alkaline.	Like lime, but not so strongly crystalline.	Like lime.	Like lime.	After long blowing has a pale flesh-color, only properly seen when quite cold. Phosphate and arsenate fuse and become violet-red.

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Table VI., B. EARTHS.	ALONE ON CHARCOAL AND IN THE FORCEPS.	WITH BORAX ON PLATINUM WIRE.	WITH SALT OF PHOSPHORUS ON PLATINUM WIRE.	WITH SODA ON CHARCOAL.	WITH COBALT SOLUTION IN THE O. F.
5. ALUMINA, $\ddot{A}l$.	Unaltered.	Dissolves slowly to a clear glass, becoming opaque neither by flaming nor by saturation. When much is added in fine powder, the glass is cloudy, scarcely fusible, and shows a crystalline surface on cooling.	Dissolves slowly to a clear glass, that is always clear. When much is added the undissolved part becomes semi-transparent.	Swells a little, forms an infusible compound, and the excess of soda goes into the coal.	After a strong blast assumes a fine blue color, the intensity of which is only properly apparent on cooling.
6. GLUCINA, $\dot{B}e$.	Unaltered.	Dissolves largely to a clear glass, which becomes milk-white by flaming, or after cooling, when fully saturated.	As with borax.	Insoluble.	Assumes a light bluish-gray color.
7. YTTRIA, $\dot{Y}$ or $\ddot{Y}$.	Unaltered.	Like glucina.	Like glucina.	Insoluble.	0
8. ERBIA, $\dot{E}$ or $\ddot{E}$.	In R. F. becomes lighter and translucent.	Dissolves rather slowly to a clear glass, which becomes milk-white by flaming, or on cooling, when perfectly saturated.	As with borax.	Insoluble.	0
9. ZIRCONIA, $\dot{Z}r$.	Infusible, but becomes unusually luminous in a powerful O. F.	Like glucina.	Dissolves somewhat more slowly than in borax, and yields an opaque glass more quickly.	Insoluble.	Assumes a dirty violet color.

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10. THORIA, Th.	Unaltered.	Dissolves in small quantity to a clear glass, milk-white on cooling, if saturated; but if it appears clear on cooling it cannot be made opaque by flaming.	As with borax.	Insoluble.	0
11. SILICA, Si.	Unaltered.	Dissolves slowly to a clear, difficultly fusible glass, that cannot be made opaque by flaming.	Dissolves in very small quantity to a clear glass. The undissolved portion becomes semi-transparent.	Dissolves with lively effervescence to a clear glass.	With a little solution assumes a feeble bluish color, becoming black or dark gray with more. The thinnest edges can be fused to a reddish-blue glass in a very hot flame.

Table VI., C. METALLIC OXIDES.	ALONE ON CHARCOAL, ETC.	WITH BORAX ON PLATINUM WIRE.	WITH SALT OF PHOSPHORUS ON PLATINUM WIRE.	WITH SODA.	WITH COBALT SOLUTION IN THE O. F.
	IN THE OXIDIZING AND REDUCING FLAMES.				
1. TRIOXIDE OF ANTIMONY, or ANTIMONOUS OXIDE, $\ddot{S}b$.	O. F. Is displaced, and spreads partly over another spot. R. F. Is reduced and volatilized, coating the coal with $\ddot{S}b$, and tinging the flame greenish-blue.	O. F. Dissolves largely to a clear glass, yellowish while hot, colorless on cooling. On coal the dissolved acid can be driven off, so that the addition of this produces no change. R. F. The glass treated only for a short time with the O. F., becomes on charcoal grayish, and clouded from reduced Sb; but this afterward volatilizes, leaving a clear glass. With tin the glass becomes gray or black according to the degree of saturation.	O. F. Dissolves with ebullition to a clear glass, only slightly yellowish while warm. R. F. The saturated glass becomes on coal, at first cloudy, afterward clear, as the $\ddot{S}b$ is reduced and volatilized. Treated with tin the glass becomes gray from reduced Sb, but on longer blowing, clear again. Tin produces a grayish cloudiness even if very little $\ddot{S}b$ is present.	Is reduced very easily on coal in either flame, but the metal volatilizes immediately, forming a white coat of oxide of antimony.	A coat of $\ddot{S}b$ moistened with cobalt solution and ignited in the O. F., volatilizes in part, but the remainder is more highly oxidized, and when quite cool appears dirty, dark green.

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Table VI., C. METALLIC OXIDES.	ALONE ON CHARCOAL, ETC.	WITH BORAX ON PLATINUM WIRE.	WITH SALT OF PHOSPHORUS ON PLATINUM WIRE.	WITH SODA.	WITH COBALT SOLUTION IN THE O. F.
	IN THE OXIDIZING AND REDUCING FLAMES.				
2. ARSENOUS OXIDE, $\ddot{A}s$.	Volatilizes below a red heat.	0	0	Is reduced on coal with evolution of arsenical fumes, which can be recognized by their strong garlic odor.	0
3. TRIOXIDE OF BISMUTH, $\ddot{B}i$.	O. F. On platinum foil fuses easily to a dark brown mass, pale yellow when cold. On coal is reduced in O. F. and R. F. to metal, and gradually volatilized, forming a coat of yellow oxide; beyond this coat is another thinner white coat of carbonate. In the R. F. these coats disappear without coloring the flame.	O. F. Dissolves easily to a clear yellow glass, which, when little is added, is colorless on cooling. With more, the glass is yellowish-red while hot, becomes yellow in cooling, and when quite cold, is opalescent. R. F. The glass on coal is at first gray and clouded, then the oxide is reduced to metal with effervescence, and the glass becomes clear. Addition of tin accelerates the reduction.	O. F. Dissolves easily to a clear yellow glass, colorless on cooling. With much the glass can be flamed to an enamel, and with still more it becomes enamel-white of itself on cooling. R. F. On coal, especially with tin, the glass changes, becoming clear and colorless while hot, but blackish-gray and opaque on cooling.	On coal it is reduced immediately to metallic bismuth.	0
4. OXIDE OF CADMIUM, $\dot{C}d$.	O. F. On platinum foil is unchanged. R. F. On coal it shortly disappears and coats the surrounding coal with a reddish-brown to dark yellow powder, the color of which is only properly seen when cold. The coal beyond the coat shows a variegated tarnish.	O. F. Dissolves very largely to a clear yellowish glass, almost colorless on cooling. When strongly saturated the glass becomes milk-white by flaming, and with still more, becomes enamel-white of itself on cooling. R. F. The glass containing oxide boils on coal; the cadmium is reduced and immediately volatilized, coating the coal with dark yellow oxide.	O. F. As with borax; the clear glass is colorless on cooling, and if saturated, appears milk-white when cold. R. F. The dissolved oxide is slowly and imperfectly reduced on coal, forming a very slight dark yellow coat, only showing its proper color on cooling. Tin accelerates the reduction.	O. F. Insoluble. R. F. On coal is immediately reduced and volatilized, coating the coal with reddish-brown to dark yellow oxide. The more remote portion of the coal assumes a variegated tarnish.	0

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5. CEROUS OXIDE, $\dot{C}e$ or $\ddot{C}e$.	Is converted by the O. F. into brick-red ceric oxide, $Ce_3 O_4$.	O. F. Dissolves to a dark yellow to red glass, similar to that of sesquioxide of iron; on cooling, the bead is yellow. With a certain degree of saturation it can be flamed enamel-like, and with more becomes so of itself on cooling. R. F. The yellow glass becomes colorless. A strongly saturated glass becomes enamel-white and crystalline on cooling.	O. F. As with borax, but the color disappears entirely when cold. R. F. The glass is quite colorless, both hot and cold, thus being distinguished from a sesquioxide of iron glass.	Insoluble. The soda goes into the coal.	0
6. SESQUIOXIDE OF CHROMIUM, $\ddot{C}r$.	Unaltered in O. F. and R. F.	O. F. Dissolves slowly, but colors strongly. With little, the hot glass appears yellow (chromic acid), but is yellowish-green on cooling; with more, it is dark red when hot, but becomes yellow in cooling, and when quite cold, fine yellowish-green. R. F. The slightly saturated glass has a fine green color, both hot and cold ($\ddot{C}r$). With more it becomes darker, or pure emerald green. Tin causes no change.	O. F. Soluble to a clear glass, reddish while hot, but dirty green when cooling, and a fine green when quite cold. R. F. As in the O. F., but the colors are somewhat darker; the same with tin.	O. F. On platinum wire dissolves to a dark brownish-yellow glass, opaque yellow on cooling (chromic acid). R. F. The glass is opaque and green on cooling ($\ddot{C}r$). Cannot be reduced to metal on coal, but remains as green $\ddot{C}r$, while the soda goes into the coal.	0
7. MONOXIDE OF COBALT, $\dot{C}o$.	O. F. Unchanged. R. F. Shrinks somewhat, and is reduced without fusing to metal, which is magnetic, and assumes a metallic lustre when rubbed in the mortar.	O. F. Colors very strongly; the glass is pure smalt-blue, both hot and cold. The strongly saturated glass is so dark blue that it appears black. R. F. As in O. F.	O. F. As with borax, but with the same quantity the color is not quite so intense, as is seen especially on cooling. R. F. As in O. F.	O. F. On platinum wire, in very small quantity, dissolves to a transparent, feebly red mass, gray when cold. R. F. On coal is reduced to a gray magnetic powder, assuming a metallic lustre by friction.	0

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Table VI., C, METALLIC OXIDES.	ALONE ON CHARCOAL, ETC.	WITH BORAX ON PLATINUM WIRE.	WITH SALT OF PHOSPHORUS ON PLATINUM WIRE.	WITH SODA.	WITH COBALT SOLUTION IN THE O. F.
	IN THE OXIDIZING AND REDUCING FLAMES.				
8. OXIDE OF COPPER, Cu.	O. F. Fuses to a black globule, soon spreads out, and is reduced on the lower side. R. F. Reduced below the melting point of copper; the reduced particles show a copper lustre, but when the blast is stopped, oxidize again on the surface, becoming black or brown. Strongly heated, it fuses to a button of copper.	O. F. Colors rather strongly; a little causes a green glass while hot, and a blue when cold. With more it is dark green to opaque when hot, but greenish-blue on cooling. R. F. Saturated to a certain degree, the glass soon becomes colorless, but on cooling is red and opaque (suboxide, Cu_2O). On coal the copper is reduced to metal, and the cold glass is quite colorless. A glass containing oxide treated on coal with tin becomes brownish-red and opaque on cooling (suboxide).	O. F. The colors are the same as with borax, but not so strong for the same amount of oxide. R. F. A rather strongly saturated glass becomes dark green, and on cooling changes at the moment of solidifying to an opaque brownish-red (suboxide). A glass containing but little, if treated on coal with tin, appears colorless while hot, but becomes red or brownish-red and opaque when cold.	O. F. Dissolves on platinum wire to a clear green glass, losing color and becoming opaque on cooling. R. F. On coal is reduced very easily to metal, which can be fused to one or several buttons by a sufficient heat.	0
9. OXIDE OF DIDYMIUM, Di or D̈i.	Unaltered.	O. F. Dissolves to a clear colorless glass. R. F. Highly saturated becomes slightly rose-colored.	O. F. as with borax. R. F. Highly saturated, the bead after long treatment appears violet-red by transmitted light.	Insoluble. The soda goes into the coal.	0
10. TEROXIDE OF GOLD, Ä̈u.	Is reduced on ignition in either flame, and can be easily melted to a button.	O. F. Reduced without dissolving; on coal can be fused to a button. R. F. The same.	As with borax.	As with borax; the soda goes into the coal.	0

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11. Oxide of Indium, $\dot{In}$.	O. F. Becomes dark yellow when heated, but on cooling is lighter again, and does not fuse. R. F. Gradually reduced and volatilized, depositing a coat on the coal. A very distinct violet-colored flame is produced.	O. F. Dissolves to a clear glass, feebly yellowish while hot, colorless on cooling, and cloudy when much is added. R. F. The glass is unchanged. On coal the oxide is reduced, volatilizes, and coats the coal again with oxide. The violet flame is perceptible notwithstanding the soda.	As with borax, but the glass when treated with tin on charcoal, becomes gray and cloudy on cooling.	O. F. Insoluble. R. F. Is reduced on coal, and the metal partly volatilizes, coating the coal with oxide, while it may partly be observed as almost silver-white globules in the fused salt.	0
12. Binoxide of Iridium, $\dot{Ir}$.	Heated to ignition is reduced, but cannot be fused.	O. F. Reduced without dissolving, but cannot be united to a button, even on coal. R. F. The same.	As with borax.	As with borax; the soda goes into the coal.	0
13. Sesquioxide of Iron, $\ddot{Fe}$.	O. F. Unchanged. R. F. Becomes black and magnetic (Fe_3O_4).	O. F. With little the glass is yellow when hot, colorless on cooling; with more it is red while hot, yellow when cold; with still more, dark red while hot and dark yellow on cooling. R. F. The glass becomes bottle-green (Fe_3O_4). On coal with tin it becomes at first bottle-green, but on longer blowing, vitriol-green (protoxide, $\dot{Fe}$).	O. F. with a small amount the hot glass is yellowish-red, but becomes first yellow while cooling, then greenish, and finally colorless. With very much it is dark red when hot, and in cooling becomes brownish-red, dirty green, and finally when cold, yellow. The colors disappear on cooling much sooner than with borax. R. F. The glass seems unchanged with a small amount of oxide; with more it is red while hot, and on cooling becomes yellow, then greenish, finally smoky-brown. With tin on coal it becomes green in cooling, and finally colorless (protoxide).	O. F. Insoluble. R. F. Is reduced on coal, yielding a gray, magnetic, metallic powder when the particles of coal are washed away.	0

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Table VI., C. METALLIC OXIDES.	ALONE ON CHARCOAL, ETC.	WITH BORAX ON PLATINUM WIRE.	WITH SALT OF PHOSPHORUS ON PLATINUM WIRE.	WITH SODA.	WITH COBALT SOLUTION IN THE O. F.
	IN THE OXIDIZING AND REDUCING FLAME.				
14. OXIDE OF LANTHANUM, La or La.	Unchanged.	O. F. Dissolves to a clear, colorless glass, that becomes enamel-white by flaming when saturated to a certain extent, and when strongly saturated becomes enamel-like of itself on cooling (Berzelius). R. F. As in O. F.	As with borax.	Insoluble. The soda goes into the coal, leaving the gray oxide behind (Berzelius).	0
15. OXIDE OF LEAD, Pb.	Minium heated on platinum foil blackens, and heated to incipient redness, changes to yellow oxide. At a higher temperature this oxide fuses to a yellow glass. On coal is immediately reduced by either flame with effervescence, and the metal gradually volatilizes, coating the coal with yellow oxide; behind this is another thinner coat of white carbonate. The coats disappear under the R. F., tinging the flame azure-blue.	O. F. Dissolves easily to a clear yellow glass, colorless on cooling, and which, with a larger quantity, becomes opaque by flaming; with still more, it becomes opaque and enamel-yellow of itself on cooling. R. F. The glass spreads out on coal and becomes cloudy; on continuing the blast, the oxide reduces with effervescence to metal, and the glass becomes clear again.	O. F. As with borax, but requiring more of the oxide to obtain a glass yellow when hot. R. F. The glass becomes grayish and cloudy on coal. With more oxide, a yellow coat is formed on the coal. With tin, the glass becomes cloudier and darker gray, but never quite opaque.	O. F. On platinum wire dissolves easily to a clear glass, becoming yellowish and opaque on cooling. R. F. Is immediately reduced on coal to metal, which afterward coats the coal with oxide.	0

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16. SESQUIOXIDE OF MANGANESE, Mn.	O. F. Infusible. In a hot enough flame both the sesqui- and binoxide are altered to manganoso-manganic oxide ($Mn_3 O_4$), yielding oxygen, and turning brownish-red. R. F. The same.	O. F. Colors very intensely. The hot glass is violet (amethyst), but on cooling becomes violet-red. An excess renders the glass quite black and opaque, the coloring being only visible by pinching the still soft glass with the forceps. R. F. The glass becomes colorless (protoxide). With a very dark glass this succeeds better on coal and with tin.	O. F. A considerable addition is requisite to color the glass, which then has a brownish-violet color while hot, and is reddish-violet on cooling, but never opaque. When the glass contains so little as to be colorless, the color can be produced by nitre (*vide* manganese, § 72, *b*). A glass containing oxide bubbles and yields gas at a high temperature. R. F. The glass very soon becomes colorless, and remains then perfectly quiet.	O. F. On platinum wire or foil, in very trifling quantity, dissolves to a clear, transparent green mass, becoming opaque and bluish-green on cooling (manganate of soda). R. F. Not reducible to metal on coal; the soda sinks in, leaving the protoxide behind.	0
17. MERCURIC OXIDE, Hg.	Is instantly reduced and volatilized.	0	0	Heated to redness in the matrass it is reduced and vaporized (also when heated alone). The vapors condense in the neck to a metallic coat, which can be united to globules by carefully tapping on the matrass.	0
18. MOLYBDENUM TRIOXIDE, Mo.	O.F. Fuses, spreads out, volatilizes, and forms at a certain distance, a yellowish, pulverulent coat, consisting of small crystals nearest the assay. The coat becomes white on cooling, and the	O. F. Dissolves easily and largely to a clear glass, yellow while hot, and colorless on cooling. A very large addition produces a glass dark yellow to dark red while hot, and opaline to a bluish-gray enamel when cold.	O. F. Dissolves easily to a clear glass, which is yellowish-green while hot, if a moderate amount is added, but becomes nearly colorless on cooling. On coal, becomes quite dark, and on cooling, is fine green, owing to its reduction to the binoxide.	O. F. On platinum wire fuses with effervescence to a clear glass, milk-white on cooling. R. F. On coal fuses with effervescence at first, but afterward the fused mass is absorbed by	0

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Table VI., C. METALLIC OXIDES.	ALONE ON CHARCOAL, ETC.	WITH BORAX ON PLATINUM WIRE.	WITH SALT OF PHOSPHORUS ON PLATINUM WIRE.	WITH SODA.	WITH COBALT SOLUTION IN THE O. F.
	IN THE OXIDIZING AND REDUCING FLAMES.				
18. MOLYBDENUM TRIOXIDE, M̈o. (Continued.)	crystals colorless. Beyond this coat is a thinner, non-volatile film of binoxide (M̈o), which on cooling is dark copper-red, and has a metallic lustre; see also § 35, *g*. R. F. The greater part sinks into the coal, and can be reduced by a good flame to metal, which is obtained as a gray powder after washing away the particles of coal.	R. F. The glass produced in the O. F. becomes brown with a certain degree of saturation, and with still more, is opaque (M̈o). In a good flame black flocks of M̈o separate, and can be very distinctly seen in the yellowish glass when it is pinched out flat.	R. F. The glass formed in the O. F. becomes quite dark dirty-green, but purer green on cooling. The same on coal. With tin, a somewhat darker green (M̈o ?).	the coal, and the greater part of the oxide is reduced to metallic molybdenum, which can be obtained as a steel-gray powder by washing away the coal.	0
19. MONOXIDE OF NICKEL, N̈i.	O. F. Unchanged. R. F. On coal is reduced to a metallic, coherent, and infusible powder, which, by friction in the mortar, assumes a metallic lustre, and is decidedly magnetic.	O. F. Colors quite intensely; a little colors the hot glass violet, but when cold, a pale reddish-brown; with more the colors are darker. R. F. The glass becomes gray and cloudy, or quite opaque, owing to finely divided metallic nickel. On continuing the blast the reduced metallic particles collect together without fusing and the glass becomes colorless. On coal, especially with tin, the reduction is more rapid, and the nickel unites with the tin to a button.	O. F. Dissolves to a reddish-glass, yellow on cooling. With a large addition the hot glass is intense red, and becomes reddish-yellow when cold. R. F. On platinum wire unchanged. On coal, with tin, becomes at first opaque and gray, but after longer blowing all nickel is reduced and the glass becomes colorless.	O. F. Insoluble. R. F. On coal is easily reduced to white metallic particles, which, after the coaly matters are washed away, follow the magnet readily.	0

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20. NIOBIC (COLUMBIC) OXIDE, $\ddot{N}b$.	O. F. Becomes yellowish, but is white again on cooling. R. F. The same.	O. F. Dissolves easily to a clear, colorless glass, becoming opaque by flaming with a certain addition, and with more becomes opaque of itself when cool. R. F. A glass which, after treatment in the O. F. becomes opaque of itself on cooling, remains unaltered.	O. F. Dissolves largely to a clear glass, yellow while hot, but colorless on cooling. R. F. With a very large addition the glass becomes brown.	O. F. With an equal volume of soda fuses with effervescence; with more soda goes into the coal. R. F. The same. It cannot be reduced to metal.	Assumes a brownish-gray color.
21. BINOXIDE OF OSMIUM, $\ddot{O}s$.	O. F. Changed into osmic tetroxide which volatilizes in penetrating and suffocating vapor, having a very irritating effect on the eyes. R. F. Reduced to a dark brown, infusible metallic powder which can be very easily oxidized to osmic tetroxide.	0	0	Is very easily reduced to an infusible metallic powder, which can be obtained pure by washing away the coal, etc.	0
22. MONOXIDE OF PALLADIUM, $\dot{P}d$. 23. BINOXIDE OF PLATINUM, $\ddot{P}t$. 24. SESQUIOXIDE OF RHODIUM, $\ddot{\ddot{R}}h$.	Are reduced at a red heat, but the metallic particles are infusible.	O. F. Reduced without dissolving, but the metallic particles cannot be united to a button even on coal. R. F. The same.	As with borax.	Insoluble. The soda sinks into the coal, leaving the metal as an infusible powder.	0

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Table VI., C, METALLIC OXIDES.	ALONE ON CHARCOAL, ETC.	WITH BORAX ON PLATINUM WIRE.	WITH SALT OF PHOSPHORUS ON PLATINUM WIRE.	WITH SODA.	WITH COBALT SOLUTION IN THE O. F.
	IN THE OXIDIZING AND REDUCING FLAMES.				
25. SESQUIOXIDE OF RUTHENIUM, $\ddot{R}u$.	Like Palladium.	Like Palladium.	Like Palladium.	Like Palladium.	0
26. OXIDE OF SILVER, $\dot{A}g$.	Reduced easily to metallic silver, which fuses to one or more globules.	O. F. Partly dissolved and partly reduced. The cold glass is opalescent or milk white, according to the amount dissolved. Metallic silver fused with borax in a clay capsule yields the same glass. R. F. On coal the glass is at first grayish, from reduced silver, but after all the silver is separated and fused to a button, the glass becomes clear and colorless.	O. F. Both the oxide and the metal produce a yellowish glass. A glass containing much oxide is opalescent on cooling, and when held against the daylight appears yellowish, against the lamp flame, reddish. R. F. As with borax.	Is instantly reduced to one or more globules of silver, while the soda sinks into the coal.	0
27. TANTALIC OXIDE, $\ddot{T}a$.	O. F. Becomes slightly yellow, but is white again when cold. R. F. The same.	O. F. Dissolves easily to a clear glass, which, with a certain amount, appears yellowish while hot, colorless on cooling, and can be made opaque by flaming. With still more the glass becomes enamel-white of itself on cooling. R. F. As in O. F.	O. F. Dissolves largely to a clear glass, which, with a very large amount, is yellowish while hot, but colorless on cooling. R. F. The above glass undergoes no alteration.	O. F. With somewhat more than an equal volume of soda fuses on coal to a bead with effervescence, and soon spreads out; with more soda sinks into the coal. R. F. The same. It cannot be reduced to metal.	After long ignition appears light gray, but on cooling assumes a feeble red color, quite similar to that of magnesia. If not entirely free from alkalies, it sinters and becomes bluish-black.

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28. TELLUROUS OXIDE, $\dot{T}e$.	O. F. Fuses, and is reduced with effervescence; the reduced metal volatilizes immediately forming a white coat of $\dot{T}e$, which usually has a red or dark-yellow border. R. F. The same. The outer flame is also tinged with bluish-green.	O. F. Dissolves to a clear colorless glass, which on coal becomes gray, from reduced metal. R. F. A glass obtained on platinum wire in the O. F. becomes first gray, and after all the tellurium is reduced and volatilized, it becomes colorless again. A coat of $\dot{T}e$ is also formed.	As with borax.	Dissolves on platinum wire to a colorless glass, white on cooling. On coal is reduced and volatilized, forming a coat of $\dot{T}e$.	0
29. THALLIOUS OXIDE, $\dot{T}l$.	Fuses and is reduced with effervescence to metallic globules, which are finally volatilized, affording a slight white coat.	O. F. Dissolves easily to a colorless glass. The cooled bead, heated far below redness, becomes superficially brown, but on strong ignition evolves little bubbles and becomes colorless. R. F. At first becomes grayish, on longer heating clear.	O. F. Gives a clear glass, becoming cloudy in a gentle blast. R. F. The glass on coal becomes gray and cloudy.	On coal is reduced to metal, evolving fumes and forming a white coat.	0
30. BINOXIDE OF TIN, $\ddot{S}n$.	O. F. The monoxide burns like tinder and is changed to $\ddot{S}n$. The binoxide becomes strongly luminous, and is yellowish while hot, but dirty yellowish-white when cold. R. F. Is reduced to metal by a continued and strong flame, a trifling coat of $\ddot{S}n$ being generally formed near the assay.	O. F. Dissolves in trifling quantity, and very slowly to a clear colorless glass, remaining clear on cooling, and not becoming opaque by flaming. A saturated and perfectly cold glass, when heated to low redness becomes opaque, loses its spherical shape, and shows indistinct crystallization. R. F. A not saturated glass is unchanged. On coal some tin can be reduced from a glass containing much oxide.	O. F. Dissolves in trifling quantity, and very slowly to a colorless glass, clear both hot and cold. R. F. This glass is unchanged on coal and on platinum wire.	O. F. On platinum wire combines with the soda with effervescence to a swollen, infusible mass. R. F. Reduced to tin on coal.	Assumes a bluish-green color, not properly seen until perfectly cold.

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Table VI., C. METALLIC OXIDES.	ALONE ON CHARCOAL, ETC.	WITH BORAX ON PLATINUM WIRE.	WITH SALT OF PHOSPHORUS ON PLATINUM WIRE.	WITH SODA.	WITH COBALT SOLUTION IN THE O. F.
	IN THE OXIDIZING AND REDUCING FLAMES.				
31. TITANIC OXIDE, $\dot{T}i$.	O. F. Assumes a yellow color, but is white again on cooling. No further change. R. F. The same.	O. F. Dissolves easily to a clear glass, which, if much is present, is yellow while hot, but colorless on cooling, and becomes opaque by flaming; with very much it becomes enamel-white of itself on cooling. R. F. A small addition yields a yellow glass; more yields a dark yellow to brown glass (sesquioxide). A saturated glass becomes enamel-blue by flaming.	O. F. Dissolves easily to a clear glass, yellow while hot, if much is present, but colorless on cooling. R. F. This glass remains yellow while hot, but reddens on cooling, and assumes a fine violet color ($\dddot{T}i$). If the oxide contains iron, the glass becomes brownish-yellow to brownish-red on cooling. On coal with tin it becomes violet, unless too much iron is present.	O. F. Dissolves with effervescence on coal to a dark yellow glass, which crystallizes on cooling, with a feeble glow. When perfectly cold the glass is grayish-white to white. R. F. The same. It cannot be reduced to metal.	Assumes a yellowish-green color, similar to oxide of zinc, but not so fine.
32. TUNGSTIC OXIDE, $\ddot{W}$.	O. F. Unchanged, unless the temperature is so high that it is reduced to binoxide ($\ddot{W}$), owing to the formation of carbonic oxide gas. R. F. Becomes black (binoxide), but is infusible.	O. F. Dissolves easily to a clear colorless glass. With a rather large addition it appears yellow while hot, and with more can be made enamel-like by flaming; with still more it becomes enamel-white of itself on cooling. R. F. With a small quantity the glass is unchanged; with more it becomes yellow to dark yellow, and yellowish-brown on cooling ($\ddot{W}$). On coal a smaller amount produces this reaction, and with tin darker colors are produced, when not too strongly saturated.	O. F. Dissolves easily to a clear colorless glass, only yellow when strongly saturated and hot. R. F. The above glass, after short treatment, is dirty-green, but becomes blue on cooling ($\ddot{W}$, $\ddot{W}$); after too long a blast it is bluish-green when cold. On coal, especially with tin, it becomes dark green ($\ddot{W}$). If it contains iron, the hot glass on the wire appears yellow, but becomes brownish-red (blood-red) on cooling, as with $\dot{T}i$ containing iron. With tin on coal the glass becomes blue, if the amount of iron is inconsiderable.	O. F. On platinum wire dissolves to a clear dark yellow glass, becoming crystalline and opaque-white or yellowish on cooling. R. F. With a little soda on coal a large amount may be reduced to metallic tungsten; with more soda the whole sinks into the coal, and a yellow compound of oxide of tungsten and soda, having a metallic lustre, is formed.	0

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33. URANIC OXIDE, $\ddot{U}$.	O. F. Infusible, but assumes a dirty dark yellowish-green color (U_3O_8) R.F. Becomes black and shows this also on friction in the mortar (U_2O_5).	O. F. Like sesquioxide of iron, but with an equal amount the colors are lighter. The strongly saturated glass can be made enamel-yellow by flaming. R.F. Like Fe_2O_3. The green (U_3O_8) glass, saturated to a certain extent, can be made black by flaming, but becomes neither enamel-like nor crystalline. With tin on coal becomes dark green.	O. F. Dissolves to a clear yellow glass, greenish-yellow on cooling. R. F. The above glass becomes dirty green, but on cooling, pure and fine green (U_3O_8). With tin on coal the green becomes darker.	O. F. Insoluble. With a slight addition of soda shows signs of fusing; with more the mass becomes yellowish-brown; with still more, the oxide goes into the coal. R. F. The same; it cannot be reduced to metal.	0
34. VANADIC OXIDE, $\ddot{V}$.	Fusible. The portion in contact with the coal is reduced and goes into the coal; the remainder assumes the color and lustre of graphite, and is trioxide of vanadium (V_2O_3).	O. F. A little dissolves to a clear colorless glass, but more gives a yellow glass, greenish-yellow on cooling. R. F. The above glass changes, appearing brownish while hot, and fine chrome-green on cooling (V_2O_3).	O. F. Dissolves to a clear glass; if not in too small quantity, dark yellow while hot, and light yellow on cooling. R. F. As with borax.	Fuses together with it, and goes into the coal.	0
35. OXIDE OF ZINC, $\dot{Z}n$.	O. F. Becomes transiently yellow, and is infusible, but strongly luminous when ignited. R. F. Gradually disappears, being reduced, volatilized and again oxidized. Part of the oxide forms a new, distinct coat on another part of the coal, being yellowish while hot, and white on cooling.	O. F. Dissolves easily and largely to a clear glass, yellowish while hot, but colorless on cooling; with more this becomes enamel-like by flaming, and with still more, becomes so of itself on cooling. R. F. The saturated glass becomes cloudy and grayish by the first blast (since some oxide is separated by the imperfect fusion), but after longer blowing is clear again. On coal is gradually reduced, volatilized, and reoxidized, forming a coat on the coal.	As with borax.	O. F. Insoluble. R. F. On coal is reduced, volatilized, and reoxidized, giving a zinc coat. With a good flame the zinc flame may even be produced.	Assumes a fine, yellowish-green color, most distinct when perfectly cold.

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CHAPTER III.

SPECIAL TESTS FOR THE ELEMENTS, NECESSARY IN VARIOUS COMBINATIONS.

(See Table VI. for a general view of the commonest reactions, p. 76.)

POTASSIUM.

56. For all pure potassium salts, except the borate and phosphate, the flame test is the best, § 42. Organic compounds, yielding a luminous, yellow flame, must first be decomposed by ignition. A very little of any sodium compound renders this test unsatisfactory, while more entirely masks the violet potassa flame, unless viewed through the cobalt glass. If little soda is present, the glass entirely absorbs the yellow color of its flame, and the violet potassa flame becomes violet-red or poppy-red, but if the soda flame is very strong it will also be visible through only moderately dark cobalt glass, as a violet flame. The potassa flame can then be best identified by using two or three thicknesses of the glass and comparing the violet-red or red flame so obtained from any potassa present with the flame obtained from a sodium salt on another wire viewed through the same thickness of cobalt glass.

Lithium salts may also conceal the potassa flame, but the effect of a faint lithia flame can be obviated also by using cobalt glass. When, however, much sodium or lithium is present and very dark cobalt glass becomes necessary, a feeble potassa flame will also be obscured, and it is then better to use some medium more transparent to the potassa flame and less so to that of soda or lithia. A solution made by dissolving one part of indigo in eight of fuming sulphuric acid, diluting with fifteen hundred to two thousand of water and filtering, is very good, but a more delicate test is furnished by a simple solution of potassium permanganate in water. If kept in a glass-stoppered prism (see below) the solution will remain sufficiently clear for many months, and when too much of a brown deposit forms on the glass it is readily removed, after emptying the vessel, by pouring in a little strong hydrochloric and sulphuric acids. The permanganate will be rapidly decomposed if in contact with a cork or even a rubber stopper, but these can be used in connection with a rather flat, rectangular bottle, always kept upright. The permanganate solution

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may be replaced by a solution of aniline night-blue in alcohol, which is then to be sufficiently diluted with equal parts of water and alcohol and afterward mixed with enough alcoholic solution of Hoffman's violet, or any reddish aniline solution, to give to the whole the same color, by transmitted light, as an aqueous potassium permanganate solution of equal depth of color. Viewed through either of these solutions the potassa flame has a red color, or violet-red if the flame is very strong and the solution only moderately dark. The permanganate solution is very opaque to the soda flame, and much more so to the lithia flame than to that of potassa.

One of the above solutions is put into a narrow, wedge-shaped bottle, Fig. 23, or "indigo prism," of white glass, with a glass stopper, well fitted. The strength of the solution should be such that the strongest possible lithium flame, obtained by igniting lithium chloride on platinum wire in the Bunsen burner flame, is totally invisible, at least through the thickest stratum of liquid; the strongest flame from sodium chloride will disappear long before the thickest part of the prism is reached. Even the feeblest potassium flame will still be visible through any part of the prism, but naturally the thinner parts will be used when not much sodium or lithium interfere. The prism may be replaced by a rectangular bottle, not square in section, but of such shape that, viewed through one face, the strongest soda flame will be cut off, while through the face at right angles to this, the strongest lithia flame will be invisible. The student must remember that the yellow, luminous flame from burning organic bodies, like wood or paper, or an ordinary gas flame may appear red throngh the above media.

Fig. 23.

Infusible or difficultly fusible substances should be finely powdered when tested for potassium by the flame, while silicates that give no reaction alone are mixed with two parts of powdered, ignited gypsum, free from potassium salts, and are then heated on wire, when the potassium is volatilized as sulphate.* Should substances be present

* The most delicate test for potassium in silicates is made by mixing the powdered substance [46] with water to a stiff paste with three or four parts of powdered calcium carbonate, see § 134, and one of ammonium chloride (both known to be free from potassium salts) and holding the paste on a platinum wire loop in the Bunsen burner flame, viewing the resulting flame coloration through the permanganate prism, unless the potassa flame is evident without it. When used for determining minerals the prism may better be replaced by sufficiently dark cobalt glass, in order not to make the test too delicate, so as to show mere traces of potassium. Poole's mixture for lithium, § 60, serves well for potassium also.

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which would injure the wire, the potassium compound must be dissolved out in water or acid, if possible, and the clear solution concentrated by evaporation and tested on wire. If not soluble, or if other injurious substances also dissolve, use the wet way, or follow § 57.

57. When more than a trace of potassium is present in any form, it can be detected by treating the finely divided substance with one part of soda and one part of borax, with a strong R. F. in a deep cavity on coal, adding a lead or gold bead if reducible bodies are present, and when such have been separated and the substance is fully decomposed, the slag is removed, powdered, boiled with a little water, filtered, the solution acidulated with hydrochloric acid, evaporated nearly dry, and the few remaining drops tested by the flame as above, through the prism. Sulphides should first be roasted.

58. If a borax bead containing nickel oxide, free from cobalt, is fused in O. F. on platinum wire with addition of a little boric acid and a considerable quantity of any potassium salt, the bead will become bluish when cold. This test is rarely, if ever, used.

Note.—*Caesium* and *rubidium* very closely resemble potassium in the reactions of their compounds, but they are so rarely met with, except in the minutest quantity, that they will probably never mislead the mineralogist, who may safely ascribe the reactions indicating potassium to that element. The spectroscope furnishes the readiest means of detecting and identifying them.

Sodium.

59. Sodium is easily detected under all circumstances by the yellow flame, § 42, invisible through the media mentioned in § 56. The student must remember that the merest trace of sodium will produce a feeble yellow color, and, therefore, a moderately strong flame, continuing for some time, alone indicates that sodium forms an essential ingredient of the substance under examination.

Lithium.

60. The carmine flame, § 42, is characteristic of lithium compounds, and is invisible through the media mentioned in § 56 if they are dark enough. Strontia and lime give red flames, the former especially resembling lithia compounds, and the soda flame obscures the lithia flame more or less completely. In presence of sodium, strontium, or calcium (lime), Chapman advises to mix the powdered substance with two parts of chloride of barium, when the soda flame at first visible will soon be followed by the baryta flame, and then

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by the lithia flame, while strontia and lime produce no coloration with chloride of barium.

If the red flame is due to lithium it will also be readily obtained by making a stiff paste of one part of the finely powdered substance with about twice as much of a mixture of one part of fluorite and one and a half of potassium bisulphate, and treating the paste on wire for the flame coloration, which will be an intense red in case lithium is present. This mixture is especially to be recommended for use with silicates; try [50].* In place of the above, Poole's mixture of two parts of ignited gypsum and one of fluorite may be used, but it presents little, if any, advantage. All of the ingredients must be well mixed and in the finest powder.

No silicates yield a strontia flame, and the lithia flame is not yielded by sulphates, although it is not confined to silicates.

Ammonium.

61. Ammonium compounds generally yield sublimates in the closed tube. The ammoniacal odor is the readiest test. If it is not perceived when the substance is heated alone, repeat the test on the powdered substance mixed with two parts of dry soda; or heat the substance in a test tube with solution of potassa. Any ammonia evolved is recognized by its odor, by the blue color imparted to a strip of moistened red litmus paper introduced into the open end of the tube (turmeric paper is colored brown), and by the white clouds of chloride of ammonium formed about a rod wet with strong hydrochloric acid and held just at the open end of the tube. Many organic nitrogenous bodies thus treated yield ammoniacal vapors, even when no ammonium compound was originally present. If there is reason to suspect this source of error, test for ammonium by rubbing the powdered substance with a drop or two of cold water and a little solid caustic potassa or a little quick-lime (made by strongly igniting calcite, or carbonate of lime, on coal in O. F).

Barium.

62. The flame test, § 42, is characteristic for barium salts. Carbonates and sulphates give the reaction well after prolonged ignition,

* Lepidolite will also give a strong lithia flame when tested alone. The red flame obtained from lithium compounds by the use of the above flux has not the pure carmine color of the lithia flame alone, but it is very characteristic under the circumstances. The student may practice on it not only with lepidolite, but also with spodumene, petalite, triphyllite, or other minerals not yielding so decided a lithia flame when treated alone.

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for which purpose the R. F. is best with the sulphate, especially if it is then moistened with hydrochloric acid and brought within the inner flame. Silicates are decomposed with hydrochloric acid, in the raw state if possible, or after fusion with soda on wire, the solution filtered, without evaporating to dryness, dilute sulphuric acid added, the white precipitate of sulphate of baryta collected on a filter, washed thoroughly with hot water, dried, and tested by the coloration of the flame. Strontium, if present, may also be precipitated as sulphate, and obscure the baryta coloration, in which case the mixed sulphates are fused, as Chapman directs, in a platinum spoon or on platinum foil, with three or four parts of chloride of calcium, the resulting mass boiled with a little water, filtered, and the filtrate, diluted somewhat more, is treated with a drop or two of potassium chromate, when a yellow precipitate will indicate the presence of barium.

Strontium.

63. The flame test, § 42, is characteristic of strontium salts, and can easily be obtained from the carbonate and sulphate, especially on adding hydrochloric acid, as directed for the barium salts, § 62. Silicates are decomposed with hydrochloric acid, in the raw state, or after fusion with soda, and the strontium precipitated and further tested as sulphate of strontia, on adding dilute sulphuric acid; see under barium. For the detection of a little strontium in presence of barium and calcium (lime) see § 131.

Calcium (Lime).

64. Lime is oxide of calcium. In the absence of obscuring compounds, the yellowish-red flame, sometimes so intense, in case especially of chloride of calcium, as to be mistaken by the beginner for the strontia flame, is characteristic of calcium salts, § 42. It can be distinctly obtained from the carbonate, sulphate and fluoride, and from the silicate *wollastonite*, the directions for obtaining the baryta flame, §62, applying here also. When the presence of calcium cannot be certainly inferred from the flame test or from the known composition of a mineral as determined according to the tables, p. 255, it must be sought for by the wet way; see § 131.

Magnesium.

65. The characteristic test is the flesh-red, or pale rose color, yielded by magnesia with cobalt solution, § 55. This is afforded by

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magnesia, its hydrate, carbonates, sulphates, and difficultly fusible or infusible silicates; the borate, arsenate and phosphate become violet. The presence of other bases may obscure the reaction, a little iron oxide rendering it pale brownish-red, and it must be remembered that the color is more intense after strong and long ignition. When not too much alumina is present the silicates may assume a color due to both alumina and magnesia. If not thus detected, the wet way must be employed; see chapter V.

Aluminium.

66. The test with cobalt solution is characteristic, § 55, but the blue color can be certainly referred to alumina only when the ignited mass has not fused and has not a lustrous surface on cooling, since a fused, blue, glassy slag may owe its color simply to dissolved oxide of cobalt. Infusible alumina compounds or such aluminium salts as leave infusible residues after strong ignition alone on charcoal, will show a true alumina reaction with cobalt solution, unless they contain substances that mask the reaction, especially the metallic oxides, or much magnesia, this last often producing a violet color with the alumina and cobalt solution. Corundum, topaz, chrysoberyl, and similar very hard minerals must be treated in the finest powder. Sulphates and certain phosphates and silicates of alumina show the reaction well. Finely powdered silica alone may assume on ignition with cobalt solution a paler, reddish or purplish blue color, and will sometimes not show signs of fusion unless very carefully examined under the lens, hence the student must prepare for this test by comparing the various reactions of alumina, silicates, and silica with cobalt solution.

If not thus found, the wet way must be employed for aluminium.

Rare Earth-Metals.

GLUCINUM (or BERYLLIUM), ZIRCONIUM, THORIUM, CERIUM, LANTHANUM, DIDYMIUM, YTTRIUM and ERBIUM are included here.

67. *a. The pure oxides* behave as described in Tables II. to VI., but their separation from each other and from accompanying bodies is to be accomplished in general only by complicated processes of wet analysis, for which the student is referred to the proper works on chemical analysis. Plattner (American edition, 1873, and German edition, 1878) gives detailed methods for special cases. In the chapter on determinative mineralogy, pp. 255 *et seq*, certain details are also given.

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b. Zirconium in dilute hydrochloric acid solution turns turmeric paper reddish-brown to orange-red, the latter color especially after drying. It may be brought into solution, when the minerals are not decomposed by the acid, by fusing them with soda and heating the mass with hydrochloric acid [12].

According to Pisani, titanic acid produces a similar color with turmeric paper, and if present must first be reduced to titanous oxide by boiling the acid solution with zinc. Molybdic solutions acidified with hydrochloric acid produce also a brown color with turmeric paper. (Fresenius.) Compare boric acid, § 113, *d*, 1.

Chromium.

68. The characteristic tests for chromium are those with the fluxes, § 46, *j*, and Tables I. to VI. If the fluxes fail, the wet way may be tried, as follows.

a. Silicates are mixed in fine powder with one and one-half volumes of soda and three-fourths of a volume of borax, fused together on coal in O. F., the glass finely powdered, covered with hydrochloric acid in a porcelain dish, evaporated to dryness, a drop or two more of the acid added, the whole warmed for a few moments, but not enough to expel all the acid, water added, the solution filtered, boiled with a drop or two of nitric acid, and then heated with excess of ammonic hydrate. The precipitate, which may contain sesquioxides of chromium, aluminium, iron, etc., is collected on a filter, washed, dried, and fused on platinum wire in O. F., or on platinum foil, with two volumes of soda and two of nitre. The resulting mass is boiled with a little water, the solution filtered, acidified with acetic acid, and treated with a little solution of acetate of lead. Any chromium present will be precipitated as yellow chromate of lead, which may be rendered very pale by the presence of sulphate or phosphate of lead. It is collected on a filter, washed, dried, and tested with borax and S. Ph. for chromium.

b. Spinel is fused on platinum wire with two volumes of soda and three of borax, the powdered bead fused with its volume of nitre, then dissolved in water, the solution filtered, acidified with acetic acid and tested with acetate of lead as in *a*.

c. All other oxidized compounds of chromium are very finely pulverized and at once treated as directed for the precipitate of sesquioxide of chromium, etc., in *a*.

Titanium.

69. *a.* The characteristic reaction is the violet color with S. Ph. and

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tin on coal in R. F., Tables II. to VI., and § 46, *f*. Much iron or tungsten obscures the reaction.

b. In presence of non-reducible oxides which color the S. Ph., the finely powdered substance is well fused with six to eight parts of bisulphate of potassa in a platinum spoon, the mass heated with very little water, just enough to remove the soluble portion, the clear solution decanted after settling, and boiled for some time with two or three drops of nitric acid and six volumes of water. If a white precipitate of titanic acid forms, it is collected on a small filter, washed with water containing nitric acid, dried, and tested with S. Ph.

c. On fusing titaniferous substances with soda, dissolving the fused mass in hydrochloric acid and boiling the solution some time with tin, the titanic acid is reduced to titanous oxide, which colors the solution pale violet, and on long boiling separates as a violet precipitate of titanous hydroxide. (Brush's Manual.)

d. If titanic oxide [56] is dissolved in large quantity in S. Ph. in O. F., on wire, and the clear, colorless bead *finely* powdered and boiled with fifteen or twenty drops (one cubic centimetre, or so) of strong hydrochloric acid in a test-tube, together with a few little shavings of tin, the acid slowly acquires a pale violet-red color, which becomes blue on cooling the solution. This color remains after diluting the acid with five or six volumes of water, but becomes pale violet or violet-red on heating. If the amount of titanium is small, the solution only acquires a pale violet color. The solution becomes turbid on boiling, as in *c*. A bead containing titanic oxide similarly treated, but with dilute hydrochloric acid (one of acid to four of water), does not color the acid after long boiling, although if left standing it very gradually acquires a faint violet color, and the precipitate found in it also becomes pale violet ; see Table VII., § 100, *d*.

Columbium (or Niobium).

70. *a*. The student will rarely have occasion to test for this metal apart from the operations of determinative mineralogy. Its general reactions are given in Tables II. to VI.

b. If a bead of S. Ph. is highly charged with columbic acid in O. F. on wire, and the powdered glass boiled with a few drops of strong hydrochloric acid and some shavings of tin, the acid gradually assumes a dirty blue or bluish-brown color and becomes turbid. If zinc is used in place of tin, the acid quickly becomes fine blue, but when poured off from the zinc and heated with five or six times as much water, it very quickly loses its blue color. If dilute acid is

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used in place of strong acid (about three cubic centimetres), the acid boiled with the powdered bead and tin does not become blue; but if zinc is used in place of the tin it rapidly becomes blue, owing to the reduction of the columbic acid to lower oxides. The blue solution very quickly loses its color when poured off from the zinc and heated with five or six times its volume of water; see Table VII., § 100, *d*.

c. To detect *columbium* in presence of *tungsten* in minerals, decompose the very fine powder thoroughly by fusion with potassium bisulphate, powder the mass very fine, warm it with water, filter, wash the residue with hot water, digest it on the filter for some time in a warm place with ammonium sulphide, filter off the liquid, wash the residue thoroughly, and test it for columbium as in *b*. The tungsten can be thrown down from the ammonium sulphide filtrate by slightly acidifying this with hydrochloric acid, after which the precipitate may be filtered out, washed, roasted, and tested for tungsten with S. Ph. and tin, § 100, *d*.

Tantalum.

71. *a*. The remarks under columbium, § 70, *a*, apply equally to tantalum.

b. Tantalic acid treated as directed for columbic acid, § 70, *b*, with strong hydrochloric acid and tin, or with dilute acid and zinc, yields no blue color, except such as may be due to traces of columbium.

Manganese.

72. There are three excellent tests for manganese:

a. The amethyst (violet-red) color, imparted by it to both borax and S. Ph. on platinum wire in O. F., which disappears in R. F., § 46, *n*, especially if the bead is treated with tin on charcoal. This is due to the reduction of the sesquioxide to protoxide. The presence of cobalt or nickel may modify the color of the manganese bead, as shown in Tables II., III., IV., and V., § 48, and these, as well as other coloring oxides, may quite obscure it.

b. When very little manganese is present, or when other coloring oxides obscure the reaction, the manganese can still very often be detected in the S. Ph. bead on platinum wire, by fusing it in O. F. and quickly touching it, while still fluid, to a small fragment of nitre (potassium nitrate), when the bead will swell greatly and assume a fine purple color, feeble traces of which can be seen, even if very little manganese is present. The color is due to the formation of potassium permanganate, and it is not entirely masked even by cobalt, unless in large quantity.

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c. The most delicate test for manganese is made by fusing the substance [2] on platinum wire, or better still, on platinum foil, with three parts of soda and one of nitre. (In difficult cases the delicacy may be yet increased, according to Chapman, by first dissolving a considerable quantity of the substance in a borax bead, which is to be powdered and treated as above.) Sodium manganate, having a bluish-green color when cold, is formed, provided the salts have been brought to perfect fusion in O. F., but an excess of manganese must be avoided, as it renders the color too dark. The presence of chromium may give rise to yellow sodium chromate, coloring the whole yellowish-green. If silicic acid and cobalt are present, the green sodium manganate may be changed to greenish-blue, or even to a blue mass in which manganese cannot be recognized. In this case use the test in *b*, and if this fails, follow the wet way.

d. Sulphides and *arsenides* must be roasted before trying any of the above tests. Metals (alloys) are first fused in O. F. beside a borax bead on charcoal, until enough of the manganese has passed into the borax bead to color it, or the bead may then be tested according to *c*. If no manganese is thus found the alloy must be treated by the wet way.

Iron.

73. The characteristic tests for iron are made with borax and S. Ph., Tables I. to VI. The magnetic properties of the metal and its magnetic oxide, Fe_3O_4 (proto-sesquioxide) are characteristic in the absence of nickel and cobalt.

Metallic iron is fusible with great difficulty and not volatile. It is obtained by the reduction with soda (especially if charcoal dust is added and a strong flame used), § 50 *et seq.*, in minute, shining, iron-gray scales, mixed with a grayish-black powder, consisting in part of metallic iron with the magnetic oxide. It is strongly magnetic, and when collected with the magnet, transferred to coal and heated alone in a gentle O. F., is quickly converted into black magnetic oxide. This treated with borax in O. F. on wire, finally gives a yellow bead. Iron oxides cannot be reduced to metal from borax by the blowpipe flame alone.

74. *a. Oxidized compounds of iron* are treated with borax on wire in O. F., the least trace of the sesquioxide (ferric oxide) rendering the bead yellow while hot, even if it becomes colorless on cooling. When there is no reason to suspect the presence of any of the rarer substances which produce similar colors, mentioned in Tables I. to VI., this reaction indicates iron with certainty. The further tests may be employed if necessary.

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Note.—Iron is a very common impurity in minerals, and even in chemical preparations; for instance, in borax. The student can determine easily whether any slight yellow color in his hot borax bead is produced by iron by dissolving a considerable quantity of the substance causing this color in borax on wire in O. F., and then treating the bead in R. F. If the bead does not now assume a decided color, dark-brown to black, blue, green, or red, and if a test with S. Ph. similarly made also fails to yield a colored bead, violet, green, blue, or red, none of the other coloring oxides can be present (in sufficient quantity to determine the character of minerals, for instance), and the color may, in nearly every case, be ascribed to the presence of a very little iron, forming an unessential ingredient in the substance. All minerals properly described as giving with borax the *iron reaction* give it strongly, making the bead dark yellow to red while hot, and at least light yellow on cooling, if dissolved in considerable quantity. They will also yield a more or less deeply colored bottle-green bead with borax in R. F. on wire, unless some other coloring oxide interferes with this part of the test.

b. Compounds containing any considerable amount of oxide of iron generally yield a *magnetic* residue when reduced on charcoal, sometimes alone, sometimes with soda, but cobalt and nickel compounds may do the same. Silicates with about 14 per cent. of iron usually begin to show magnetism when reduced alone.

c. Iron and cobalt. A borax bead containing iron and very little cobalt becomes in O. F. on wire yellowish-green to green, both while cooling and when cold; with more cobalt it may be bluish-green or even blue when cold, but will still be green or greenish-blue while hot, these colors being due to the yellow of the iron with the blue of the cobalt. When the cobalt predominates very largely the iron reaction cannot be obtained.

d. Oxide of iron with manganese (and cobalt). The presence of manganese may conceal more or less completely the iron reaction, causing a yellowish-red to red bead. In this case the borax bead is treated in a strong R. F. on wire, or, in presence of much manganese, with tin on charcoal, long enough to reduce all manganese to the colorless protoxide, whereupon the color of protoxide of iron, § 46, *h*, will appear, dark yellowish-green (bottle-green) on wire, vitriol-green with tin on coal. The student must remember that a bead containing a *large* excess of iron will become quite black after this reduction with tin, a case not likely to occur, however, under these circumstances.

Iron can be detected in presence of comparatively large quantities of manganese by using a bead of S. Ph. in place of borax. The manganese oxide does not color the S. Ph. so intensely as it does the borax, and hence either the yellow color of the iron oxide may still predominate on treatment in O. F., or else the iron oxide will pro-

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duce its predominant color with the S. Ph. in R. F. on wire, viz., smoky reddish-brown.

If, in addition to the manganese, a little cobalt is also present, with not too little iron, the reduced borax bead will be bluish-green to greenish-blue. If too much cobalt is present, together with manganese, the wet way will be found most convenient for the detection of iron, unless it is possible, by a reduction with soda, to obtain some metallic iron, which can be separated with the magnet and identified as in *c*, even if accompanied by some cobalt.

Iron can, indeed, be detected in oxides, even in presence of much cobalt and manganese, by arsenicizing the oxides, § 183, with addition of iron-free nickel oxide, if this is needed to make the resulting arsenides fusible. The iron is then found as in § 76, *d*. The wet way is, however, simpler.

e. Reducible oxides being also present in a borax bead, the iron would be best detected by making a reduction assay, with addition of lead, or gold, if needed, § 44, *d*, so as to remove from the borax all except non-reducible oxides. The still soft borax bead is then pinched out with the forceps and some of the clean glass examined for iron according to *a*, *c*, or *d*.

f. If *chromium oxides* are present with oxide of iron, the iron may often be detected by the properties of its magnetic oxide, obtained by a gentle reduction of the powdered substance on coal. Should this test fail, fuse the powder with three parts of nitre and one of soda on platinum wire in O. F., boil the powdered mass with a little water, wash the insoluble residue thoroughly on a filter with hot water and test it for iron with borax.

g. Oxide of uranium being present, if no magnetic oxide of iron can be formed, see *f*, fuse the powdered oxides with potassium bisulphate in a platinum spoon, dissolve the resulting mass in water, add ammonium carbonate in excess and test the insoluble residue for iron with borax. Should the original powdered substance be soluble in hydrochloric acid or aqua regia, its solution can be treated at once with the ammonium carbonate, omitting the fusion.

h. Titanic or tungstic oxide, in more than traces, give with oxide of iron in S. Ph. a brownish-red bead in R. F.; compare Table VI., C., and § 46, *f*.

i. Sulphides and *arsenides of iron*, with or without a little arsenide of cobalt or nickel, should be very thoroughly roasted and then treated as directed above for oxides, simple or mixed.

k. Arsenides containing iron with much cobalt or nickel; see § 76, *d*.

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l. Alloys. If difficult to fuse these are treated with a borax bead on coal in O. F., until enough of the metals have been oxidized and dissolved; if easily fusible, containing lead, tin, etc., a good R. F. is directed rather on the flux than on the alloy, and in either case the borax bead is then thoroughly reduced, beside lead, if necessary, § 44, *d*, to remove all reducible metals, leaving a borax glass which is examined as in *e*. If the alloy is exceedingly infusible try to alloy it with a little lead or other metal so as to render it fusible, or very finely divide it and oxidize it by gentle ignition on charcoal in O. F., so that the oxides may be tested with borax. If these means fail the wet way must be tried.

For Metallurgical Products see Chapter IV.

Cobalt.

75. The characteristic test for cobalt is made with borax and S. Ph., Tables I. to VI.

Cobalt is obtained by the reduction test with soda on coal, § 50 *et seq.*, in minute, shining, grayish-white scales or grayish powder, attracted by the magnet. This is darkened by oxidation after gentle ignition in O. F. on coal, yields no coat and dissolves in borax in O. F., giving a blue bead.

Cobalt is not reduced from the borax bead alone, but a little of it may be reduced from a saturated bead and alloyed with other reduced metals, if present.

76. *a. Mixed oxides,* with or without reducible metals, are treated as directed under iron. Iron or chromium alone, among the commoner elements, can conceal the blue color of the borax bead reduced with tin or lead, on charcoal, and then, in case of iron, only when so little cobalt is present that the bead shows no trace of blue after careful reduction with tin, being diluted with fresh borax, if necessary, to remove the black color due to excess of iron. When very little cobalt is present with much iron, the blue of the cobalt may not be perceptible in union with the vitrol-green of the iron, unless a comparison bead is made containing about as much iron with no cobalt.

b. Sulphides (with or without a very little *arsenic*) are thoroughly roasted and tested as above.

c. Selenides are first roasted on charcoal and then treated with R. F. beside a borax bead, in which the cobalt may be afterward detected, as above.

d. Arsenides (compare § 181) are fused in a shallow cavity on coal with a moderate R. F., until the arsenic fumes cease, when a

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small piece of borax glass is added, and the O. F. directed for a short time on the borax. Any iron arsenide present will be first oxidized, and if the blast is stopped as soon as the metallic button of arsenides appears bright, while still melted (and it will be bright at once if no iron is present), the iron can be detected in some of the separated glass by oxidizing this, with a little more borax if very dark, on platinum wire in O. F. The borax bead will then show the yellow color indicative of iron, or the green color due to a mixture of some blue from cobalt with the iron in the borax, if the operation had not been stopped at the right moment. When the button of arsenides is again oxidized with fresh borax the glass will be colored blue, since the cobalt arsenide will next be oxidized and dissolved.

After the cobalt is removed there will remain, if nickel (and copper) are present, a button of the arsenides, which will now no longer remain bright while melted, as it did in the presence of the cobalt arsenide, but will begin to show a film of nickel arsenate. The blast should be stopped now and the button again oxidized beside fresh borax, to which it will at first impart the reddish-brown nickel color. Any copper present can be found in the remaining button (by means of S. Ph.) as directed in § 88, *f*, 1, and *d*, under nickel and copper alloys.

The beginner will not find it easy to follow accurately the above changes in the appearance of the melted button of arsenides, especially if it is small, but the progress of the operation can always be easily followed if the first treatment of the borax with O. F. is continued for a very short time only, stopping as soon as the glass appears to have dissolved anything. The glass is then pinched or drawn out to examine its color, and further tested on wire in O. F., after dilution with borax, if necessary. The treatment of the button of arsenides is continued with fresh borax on coal, the borax being from time to time drawn out and examined, on wire if necessary, and after dilution with borax if too dark, while new borax is substituted for the old whenever the glass becomes too deeply colored to allow of certain judgment as to its true color. In this way any iron, cobalt and nickel can be conveniently determined with great certainty, by the successive colors as described in the first part of this division, *d*, while the copper which may remain, either with or without nickel, can be detected by § 88, *f*, 1. The student should practice this on a suitable sample [64].

Combined with the arsenicizing (with previous roasting if need be), § 183, this process furnishes a very delicate test for iron in presence of much cobalt, or the reverse.

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e. Alloys are generally treated as directed for iron, but to detect a little cobalt in nickel the finely divided alloy is fused in a moderately deep hole in charcoal in the R. F. with its volume of powdered metallic arsenic and the resulting arsenides tested for cobalt with borax on coal as above.

For Metallurgical Products see Chapter IV.

NICKEL.

77. The characteristic tests for nickel are made with borax and S. Ph., Tables I. to VI.

Nickel, as obtained by the reduction test with soda, § 50 *et seq.*, closely resembles cobalt, but with borax in O. F. it yields its characteristic reddish-brown bead. It is easily reduced from the borax bead either on wire or coal, but being hard to fuse is not obtained in one or two buttons, but separates in fine particles, which gradually collect to grayish masses around the wire and leave the bead colorless after long heating. It is not reduced from S. Ph. on wire, but can be reduced from it on coal beside tin, which alloys with and collects it, leaving a colorless bead.

78. *a. Oxides of non-reducible metals which color the fluxes*, as cobalt, iron, manganese, etc., are tested for nickel by dissolving them in borax on wire in O. F., and then treating the bead in a strong R. F., when the reduced nickel, if in considerable quantity, will render the bead cloudy (grayish, if the other coloring oxides are reduced to feebly coloring oxides). Since, however, it may be necessary in doubtful cases to further identify the nickel, it is in general best at once to reduce the bead containing the dissolved oxides beside a gold button (of fifty to eighty milligrammes in weight, according to the probable amount of nickel present). The reduced, metallic nickel will be collected by the gold, and can afterwards be detected by treating the gold beside a previously made bead of S. Ph. on coal in O. F., to which the oxidizing nickel will impart a yellow color when cold. If very much cobalt was present a little of it may also be reduced, giving to the S. Ph. bead at first a bluish color, but with fresh S. Ph. the pure nickel reaction can afterwards be obtained. If copper was present it will also be found in the gold, and then the S. Ph. bead will show a green color when cold, due to the yellow produced by the nickel oxide and the blue of the copper oxide in the S. Ph. bead. If very little copper was present the bead may be only decidedly green while hot, but nearly pure yellow on cooling. In any case the presence of the copper can be confirmed as in § 88, *d*, or *f*, 1.

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If oxides of lead, bismuth, etc., are present, as well as non-reducible oxides, follow *c*, below.

b. Nickel oxide containing copper oxide in sufficient quantity to form a fusible alloy of the two metals can be tested as above, by reduction of the two from borax, but no gold need be added ; nickel in the alloy will be shown by the green color of the S. Ph. bead.

c. Nickel oxide with reducible oxides of more or less volatile metals, with or without copper oxide.

1. A rapid method, by which any considerable amount of nickel can be detected, consists in reducing the oxides with borax on coal (adding test-lead, unless the substance already contains considerable lead), separating the greater part of the lead, together with any bismuth, antimony, etc., by means of boric acid, § 88, *f*, 2, and when little lead is left, heating the residual alloy alone on coal with the point of the inner flame, until an infusible mass of nearly pure nickel remains, which can be tested with S. Ph. for the yellow nickel glass. If copper is also present, the separation of the lead, etc., may be entirely effected by the boric acid and both nickel and copper found in the residual button, as in *b*.

2. A neater method for rather small quantities of nickel, in absence of copper, consists in reducing the original oxides with borax, treating the resulting alloy alone with the point of the inner flame, until most of the lead, bismuth, etc, have been expelled, alloying the remaining button with about one-half its weight of gold (and as much test-lead, if the substance contained no lead) by fusion beside borax in R. F., removing the lead by means of boric acid and testing the gold for nickel as in *a*.*

3. In presence of copper proceed in the same way, but omit adding the gold before treating the copper residue with the boric acid.

d. Arsenides ; follow § 76, *d*.

e. Sulphides, with or without a little arsenic, are thoroughly roasted and the oxides tested as in *a*, *b*, or *c*.

f. Alloys. If fusible they are first treated alone on coal in R. F., and if any coat is formed the R. F. is continued until as much as possible of the volatile bodies has been expelled. (In case of lead or bismuth, boric acid may be used to aid the separation.) The residue is then fused beside borax in a cavity on coal in R. F., to ascertain whether it contains any iron or cobalt, and when it ceases to yield anything further to the borax in R. F. the metallic button is freed

* The gold button is purified afterward by cupelling it with test-lead, p. 112, and separating the last traces of lead by fusion beside boric acid, if this is deemed necessary.

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from the glass by careful hammering between paper and is tested for nickel (and copper) with S. Ph. as already described.

Infusible alloys, containing chiefly iron, are treated as in § 74, *l*.

For Metallurgical Products see Chapter IV.

Zinc.

79. *a.* Zinc is reduced B. B. alone from many of its compounds and from all of them by the aid of soda, but it volatilizes entirely unless some other metal is present, p. 71, and forms a coat, § 35, *c*. Oxide of zinc is not reduced in a pure O. F. on coal, compare Table VI., although a very short treatment with R. F. may volatilize a not inconsiderable quantity of zinc, since its oxide is slowly reducible by the R. F. alone.

The most characteristic test (applied at once to the substance, or else to the coat) for zinc is that with cobalt solution, § 55, which fails only in presence of too much other coloring substances or bodies forming fusible blue glasses or slags with cobalt oxide. The transient yellow color of hot zinc oxide is partly characteristic; Table VI.

b. Oxidized compounds are tested at once on coal, unless it is necessary, on account of other substances present, first to form a zinc coat and to test this. The coat is formed by heating the powdered substance with a strong R. F. in a moderately shallow cavity, the addition of a little soda with half as much borax greatly promoting the formation of the coat in difficult cases, especially in presence of silicates or much oxide of iron, alumina or earthy matters. The coat is allowed to cool, moistened with a drop or two of cobalt solution and gently dried and strongly ignited in O. F. If very little zinc is present a drop or two of the cobalt solution is spread on the coal where the coat may be expected to form, and is gently dried, after which the R. F. is employed to form a coat, and the heat of the outer edge of this flame will ignite the zinc coat with the dried cobalt solution so as to produce the green coloration. The lens must very often be used to detect the green color, which may be only found here and there on the coal, sometimes almost under the assay. Compare *d, Note.*

c. Sulphides should generally be roasted and treated as above, but the roasting is not always necessary.

d. Cadmium, lead and *bismuth,* if present, will volatilize and form coats with the zinc coat, often concealing it entirely; but if these coats when quite cold are moistened with the cobalt solution, gently

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dried and carefully ignited in O. F., the green zinc and cobalt compound will appear, while the other oxides will be reduced by the glowing coal and volatilized. If very little zinc is present apply the cobalt solution first, as in *b*.

NOTE.—*Antimony*, if present (as shown by the tests, § 35, *b*,) in a substance, must always be expelled by treatment with a moderately strong O. F. on coal, before using the R. F. to make the zinc test. Sometimes it may be well to further remove any residue of oxides of antimony from the zinc coat itself by heating the coat carefully with the O. F., and in any case the cobalt solution must not be applied so long as any oxide of antimony is present, for it may yield a green coat even in the absence of zinc.

e. Tin yields with cobalt solution a reaction so similar to that of zinc that a little of the latter cannot generally be detected by this test in presence of the tin. Some alloys, however, such as bronze or bell metal, containing copper as well as tin and a little zinc, if heated for a very short time in R. F. on a coal previously moistened with cobalt solution and dried, as in *b*, may show a distinct yellowish-green coat before the bluish-green coat characteristic of tin appears. Even mixed oxides of tin and zinc will sometimes do the same, the zinc being much more volatile than the tin.

In all cases of doubt as to whether the green color is due to the presence of zinc or tin, the beginner must make comparison tests with the coats obtained from the pure metals, until he is familiar with the yellowish-green of the zinc coat with the cobalt solution and the bluish-green of the tin coat.

NOTE.—It is occasionally possible to detect a little zinc in the presence of much tin by forming a coat of the oxides of both metals on coal, scraping off this coat and heating it with a little charcoal dust in a narrow, hard glass tube, with a very strong O. F. Zinc volatilizes and forms a slight zinc oxide coat, yellowish while hot, white when cold. The test is often available, but is not to be regarded as certainly indicating the absence of a little zinc when no such sublimate is obtained.

f. Alloys are simply treated alone on coal in R. F., until a coat forms, which is tested with cobalt solution, as above.

g. In doubtful cases try the wet way.

For Metallurgical Products see Chapter IV.

URANIUM.

80. *a.* The characteristic tests are afforded by the fluxes, § 46, *i*, and Tables II. to VI., but the presence of other non-reducible, col-

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oring oxides, especially of iron, vanadium and chromium, will interfere more or less with this method of testing for uranium.

b. In presence of much *iron* acidify the ammoniacal filtrate from the insoluble residue of ferric hydrate (hydrated sesquioxide of iron), § 74, *g*, with hydrochloric acid, treat with solution of potassa in slight excess, collect the yellow precipitate of uranic hydrate on a filter, wash it and test it with S. Ph. on wire.

c. In presence of *reducible oxides*, like copper or lead, make a reduction assay with borax and soda, adding lead, if necessary, to collect the reduced metals, powder the clean slag, dissolve it in a little warm hydrochloric acid with one-third of nitric acid, dilute with water and proceed as in *b*.

Vanadium.

81. *a.* If the reactions with the fluxes, Tables II. to VI., fail, owing to the presence of other coloring, non-reducible oxides, vanadates free from silicates are fused in fine powder with two volumes of soda and two of nitre in a platinum spoon, the resulting mass treated with water, the clear solution decanted from any residue into a porcelain dish, acidulated with acetic acid, warmed, and a little powdered acetate of lead added. If a precipitate is formed it may consist of pale yellow vanadate of lead, which will settle if the solution is strongly heated. It can then be tested with S. Ph. and borax. The presence of any arsenate, phosphate or sulphate, would cause the precipitation of the corresponding salts of lead, in which case they must first be treated on coal in R. F. to remove the excess of lead. Chromate of lead, if present, will render the S. Ph. bead yellowish-green in O. F. Silicates are decomposed as directed in § 68, *b*, and the fused mass treated with nitre, etc., as above.

b. A bead of S. Ph. somewhat highly charged with vanadic acid in O. F. on wire, powdered and shaken with a little dilute hydrochloric acid (one of acid to four of water), and a strip of zinc gives a green solution; this slightly warmed often becomes greenish-blue; on further heating it finally becomes pale lavender-blue. If now poured off from the zinc and heated it quickly turns green again. With tin shavings in place of zinc the cold solution is green when agitated, becoming greenish-blue on slight warming, and green again when heated strongly; see Table VII., § 100, *d*. If the S. Ph. bead is first reduced beside tin on coal the solutions behave with zinc and tin as before, but show no blue color before being heated.

Indium.

82. For general reactions of indium see Table VI. Although

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indium occurs in exceedingly small quantities only, yet it is not unusually present in zinc blendes (sphalerite). The blende is decomposed by heating with strong hydrochloric acid, with addition of nitric acid from time to time, until the separated sulphur is free from undecomposed blende. The nitric acid is expelled by evaporation with slight excess of hydrochloric acid, the solution diluted with water, filtered, made rather strongly acid with hydrochloric acid, if not already so, saturated with hydrosulphuric acid, the precipitated sulphides of lead, copper, arsenic, etc., filtered out, the solution boiled with strips of sheet zinc, until it evolves scarcely any hydrogen, while there remains still some undissolved zinc. From this the spongy coating of reduced, metallic indium, with basic zinc salts and reduced copper, lead, etc., from the impure zinc, is scraped off, dissolved in hydrochloric acid, again separated from impurities by means of hydrosulphuric acid and again precipitated by boiling with *pure* zinc. The spongy indium, with basic zinc salts, is again dissolved in hydrochloric acid and again precipitated, this time simply by allowing its acid solution to stand for several hours over a little pure zinc. The spongy metal thus obtained is fused under potassium cyanide in a porcelain crucible, whereby it is obtained in a nearly pure state.

SILVER.

83. *a.* Silver is obtained by the reduction test, § 50 *et seq.*, in globules, as a white, malleable, easily fusible metal, which remains bright during and after treatment in O. F. on coal, affording a slight brownish coat only after rather long heating. The coat is not always easily obtained and is not used as a characteristic test.

Oxidized compounds of silver are quickly reduced to metal B. B. alone on coal in either flame; sulphides, antimonides, and arsenides yield silver after longer or shorter treatment with O. F. alone; chlorides are reduced gradually in R. F. It is better to add a little soda to all of the above substances, except oxides, while borax is also beneficial in the presence of non-reducible oxides or much earthy matter. With regard to getting the true color of the silver globule obtained by the reduction, see especially § 50, *b*. If a little lead, bismuth, or antimony is present the silver button may afford a reddish coat, § 38, *b*. This color is particularly well observed if the silver is heated on a surface of bone-ash.

b. The best test for *silver in presence of reducible metals*, like copper, lead, etc., or when very little silver is present, is made by fusing the finely divided substance with five to ten parts of test-lead

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(unless enough lead is already present), and one part, or a little more, of powdered borax glass with R. F. in a deep cavity on coal, making, in fact, a reduction assay precisely as described in § 151, without using the paper and with, perhaps, less care than the quantitative assay requires. As soon as all of the silver has been reduced and collected by the lead, about two minutes, the R. F. is altered to a pointed O. F., which is directed upon the quietly fusing lead, until it has been bubbling and boiling actively for a short time. This is done to remove all sulphur, antimony, or arsenic. The lead is then poured out upon the anvil, freed from slag, if necessary, and cupelled. The cupel is made by pressing some washed bone-ash quite tightly into a cavity in the coal about one-fourth of an inch deep and five-eighths of an inch wide, with the broad end of the agate pestle, leaving the bone-ash surface slightly concave and nearly even with the coal. The bone-ash is then slowly heated to redness in O. F. to remove all moisture, the lead is placed on the cupel and the O. F. directed upon it, the heat being made intense enough to cause the fusing oxide of lead to sink into the bone-ash, so as to leave finally the pure silver button, § 153. If the lead button is too large for this, make first a concentration, § 152, and then a fine cupellation. If much nickel or copper is present consult § 159, *b*.

The cupellation may sometimes yield only gold containing silver, which is to be treated as in § 92.

c. Alloys. These are treated as described under the quantitative assays of alloys, § 159, but with less care.

Mercury.

84. *a.* Mercury is so volatile that it is not often obtained in the metallic state in blowpipe operations, unless these are conducted in glass tubes, wherein the vapors of mercury can condense. It is, however, sometimes obtained in the metallic state as a trifling grayish, very volatile coat on coal, when its oxides or sulphide, or amalgams, are rapidly heated in somewhat large quantity.

The characteristic test for all mercury compounds, except amalgams, is made by mixing them [18], in a fine state of division, with at least three volumes of soda, which has been previously dried by being heated nearly to fusion on coal, or otherwise, and then finely powdered. The mixture is heated in a closed tube, the inside of which must be kept clean. The lower end of the tube where the powder lies is heated in O. F., or with the alcohol lamp, gently at first and then slowly to redness. If much moisture condenses in the tube after the first very gentle heating it should be wiped out, as

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moisture greatly interferes with the test. If there is very much sulphur it is well to put a little more dry soda above the mixture in the tube. Metallic mercury will sublime and form a lustrous, grayish-white, metallic mirror, which must be observed in strong, *reflected* light, being about one-half of an inch or more above the fused soda. If very little is present a few scattered globules only may condense within the tube, at some distance from the soda, forming only the faintest film, but they can always be detected and identified as mercury, after a little experience, by examining the tube through the lens, holding it in a strong light, so that the light may be reflected from the dots of mercury. These very characteristic dots soon become quite familiar, but the student should also practice on exceedingly small quantities. Some glass contains much oxide of lead and this is very apt to be reduced by the flame, but the dark, mirror-like film thus produced on the outside of the tube is confined to the end exposed to the flame. If the sublimate of mercury is at all abundant it can be collected to larger globules by rubbing with a stick, feather, or paper roll, and these can be shaken out upon paper.

b. By introducing into the tube a bit of gold leaf on the end of an iron wire, the merest trace of mercury sublimed will be shown by the whitening of the gold.

c. Amalgams are tested alone, or with a little dry soda, if sulphur or selenium is suspected, in the closed tube, the heat being *very gradually* raised to redness.

Lead.

85. Lead is obtained very easily from most of its salts and its oxides by a reduction test, soda being used only to facilitate the process in most cases, see § 86, *a*, although with the phosphate of lead it is necessary. Lead is recognized as being a light bluish-gray metal, malleable, very fusible, and yielding a yellow coat, § 37, *a*, even in a gentle O. F. on coal. It imparts to the inner flame an azure-blue tinge and is entirely volatilized by long heating.

86. *a.* The characteristic tests for lead, apart from its physical properties, are the yellow coat and the azure-blue flame; the latter (which is no indication of lead, unless accompanied by the yellow coat) is yielded both by the metal and the coat of lead oxide, although in a much less degree by the coat. The flame coloration serves to identify lead, in not too small quantity, in the presence of bismuth; in the absence of the latter, which is fortunately easily

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discovered, § 90, *b*, the lemon-yellow coat alone is, to the experienced operator, abundant proof of the presence of lead, without insisting upon the appearance of the blue flame, although this is not very difficult to obtain from a pure lead oxide coat. Cadmium oxide in large quantity (as well as the sulphides of arsenic, if incautiously driven off without oxidation, so as to form a yellow or orange coat) might lead the beginner into error, but the arsenic sulphides would be oxidized to white arsenous acid if properly treated, § 35, *a*, and, in any event, if formed as a coating, are driven away again by O. F. before the coal begins to glow, while the yellow lead oxide coat disappears rather slowly, even under a strong O. F.

Since selenium comounds, easily known by the odor, § 38 , *c*,may, if present with bismuth, afford the phenomenon of a yellow coat with the blue flame, such a combination might prevent the detection of lead, unless it was present in sufficient quantity for the test *b*. Care must be taken not to confound with the azure-blue lead flame the blue flame of burning carbon monoxide, yielded by the coal alone.

The yellow coat is best obtained from oxidized compounds by using the R. F., and the student should consult §§ 37, *a*, and 36, *c*, with regard to certain white coats from lead compounds. It is better to add soda in testing sulphides, chlorides, etc., for lead, and a little is useful also with silicates. Borax forms a useful addition in presence of much earthy matter or metallic oxides difficult to reduce.

b. A very delicate test for lead is made by treating the substance with a moderately reducing flame on a long coal with somewhat more than its volume of a mixture of equal parts by weight of potassium iodide and sulphur, as proposed by von Kobell for bismuth, § 90, *b*. The substance should be in fine powder, or if not brittle in filings. A rather volatile coat of yellow lead iodide is formed *at quite a distance from the assay*. It may sometimes be necessary to place more of the iodide mixture on the already heated substance and repeat the operation, but when little lead is present the first signs of the yellow lead iodide coat must be watched for, lest it be covered by the white potassium iodide or sulphate, which is volatilized by long heating.

A long coal is necessary, because the yellow lead iodide forms at a distance from the assay. It may easily happen, when much lead is present and the blast is continued for rather a long time, that some of the ordinary yellow lead oxide coat will form *between the assay and the iodide coat.*

Precautions.—Although this is the most delicate test for lead, in presence of other volatile bodies, short of reduction with soda, the following points must be

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noticed. Large amounts of mercury, arsenic, or antimony may either yield coats resembling the yellow lead iodide (or red bismuth iodide, § 90, *b*,) coat, or may cause white coats readily concealing it. If present, the greater part of them must be expelled by a gentle O. F., before using the iodide mixture. Cadmium too, in excessive quantity, interferes with the test. It is to be removed in great part by a very gentle R. F.

Bismuth not only yields its characteristic red iodide coat, § 90, *b*, but very often yields in addition a yellow coat, nearly or quite as far from the assay as a lead iodide coat would form. This coat is apparently also an iodide of bismuth, and although the careful observer will have no difficulty in detecting lead in bismuth oxide, containing three times as much bismuth as lead, the test soon ceases to be reliable beyond this limit, nor can the bismuth be removed by any but wet methods of analysis.

c. The delicacy of the potassium iodide and sulphur test for lead is greatly increased by treating the substance in an open tube, just as directed for bismuth, § 90, *c*. The yellow lead iodide sublimate forms at about one-half to two-thirds of an inch above the heated assay. It is easily distinguished from the sulphur sublimate just above it, because the latter shows signs of having fused. The test will show one-fourth of one per cent. of lead oxide in otherwise pure zinc or tin oxides, or in these mixed with one-half their weight of cadmium oxide; but it is not to be successfully undertaken without first studying the appearances of the sublimates by test experiments. One per cent. of lead is detected with great ease. The test is also the best blowpipe test for little lead in presence of bismuth, but a very delicate blowpipe test for lead in combination with much bismuth is at present wanting.

d. When it is necessary to test for lead in the wet way follow § 126.

For Metallurgical Products see chapter IV.

COPPER.

87. Copper is quite easily obtained from its oxidized compounds by a reduction assay with soda, § 50, *et seq.*, in one or more buttons, or if little copper is present, in minute scales or grains. The surface of the metal is often dull after this reduction, but when rubbed shows the copper color. The metal is malleable, rather easily fusible, in small quantities, and yields no coat even in the hottest flame, not being perceptibly volatile. While melted it is bright and shows a peculiar greenish hue; on cooling its surface is ordinarily a little dull, from a coating of suboxide. Heated in a gentle O. F., not to fusion, copper is superficially oxidized and becomes dark. It imparts an emerald-green tinge to the inner flame when highly heated.

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As regards the purification of the copper button with borax see § 50, *b*. A number of copper sulphides and arsenides can be reduced to quite pure copper on coal alone, using alternately the O. F. and R. F., but it is best to first roast such substances, as well as arsenates. The addition of soda is always beneficial, while borax may well be added also in presence of much earthy matter, quartz, oxides of iron, or non-reducible oxides. In any case, all that has been stated in § 50 *et seq.* must be attended to.

The characteristic tests for copper are the appearance and physical properties of the reduced metal; also the tests in the following paragraphs especially, and those with the fluxes, § 46, *l*, and Tables I. to VI.

88. *a*. The most delicate test for copper, under suitable conditions, is made by moistening the finely divided substance on coal with a drop of strong hydrochloric acid, drying it *very gently* before a small O. F., and bringing the dried mass well within the point of a rather spreading, not too luminous R. F. Chloride of copper is volatilized, giving a beautiful, intense azure-blue flame, § 42, 5, *a*. When much copper is present this may be succeeded by the emerald-green oxide of copper flame, after the chloride has been driven off. If the substance is not readily attacked by the acid it is well to drop the acid on the heated powder.

Sulphides, etc., which fail to show the test at once may first be roasted. Alloys should be treated in filings. If the substance is not readily attacked by hydrochloric acid alone it may first be heated with nitric acid; this is useful with many alloys, as of silver, and with sulphides, etc. A drop or two of mixed hydrochloric and nitric acid may also be tried; with gold or platinum for instance. The substance may also, if not readily attacked by the above acids, be first heated with them in a porcelain dish, the acid gently evaporated, and the residue tested on coal with a drop of hydrochloric acid. This plan is especially applicable to silicates and slags, if at all attacked by hydrochloric acid.

The flame test will show one or two hundredths of a per cent. of copper very readily in substances attacked by any of the above acids. It will show one-tenth of one per cent. in otherwise pure sodium chloride. Lead oxide containing over one per cent. of copper can also be tested in this way, if not too highly heated, because the copper chloride flame appears more readily than the blue flame from the lead chloride. Arsenic and selenium may interfere with the test, but they can be sufficiently expelled by preliminary treatment in R. F. to admit of the certain detection of any considerable amount of

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copper. When there is reason to suspect the presence of other substances giving flames that mask or too closely resemble the copper flame, the substance, if an alloy, may be treated according to *f*; if not an alloy, it may be subjected to a reduction test, after preliminary roasting, if need be. The reduction will either yield copper alone, or an alloy. When the metal obtained by the reduction test is exceedingly small in quantity, it may be washed entirely free from soda (and borax) and the remaining metallic grains gathered on a bit of paper and tested, after burning the paper on coal, with hydrochloric acid. This reduction method is also to be resorted to in case of substances not attacked by the acids enumerated above, since it cannot fail to furnish metallic copper or one of its alloys. The student is advised to make good use of the flame as a preliminary test for copper, since it is very easily made, and the variations above described will never be necessary when any considerable amount of copper is present; with the exception, perhaps, of the use of nitric and hydrochloric acids together when testing gold, platinum, and similar metals. He should be warned, however, that it generally shows very small quantities of copper very distinctly, and before concluding that there must be much copper present it will often be well to try one of the following tests, especially when that question is to be decided.

b. Substances suitable for such treatment may be dissolved, in large quantity if necessary, in S. Ph. on platinum wire in O. F. and the bead touched against a little sodium chloride, after which it is again fused in the inner flame, which will be tinged blue, as in *a*, if copper is present. This test is not quite so delicate as the one in *a*, but it will show very minute quantities of copper, and is especially useful in case of substances not decomposed by acids, silicates, and slags, when a reduction test is not convenient.

c. Oxidized copper compounds can always be tested by reduction with soda and borax (with addition of a little lead, if there is very little copper, and at the same time little or no lead already present) and the resulting alloy tested by the appropriate method among those described under *f*, especially *f*, 2. This test will show one-tenth of one per cent. of copper in lead oxide when two or three ivory spoonfuls of the substance are employed, enough soda and borax being added to secure a good fusion. If very small quantities of other reducible metals, or none at all, are present, the lead may be omitted and the reduced copper tested as in *a*. This test, in absence of other reducible metals, will gives scales of copper when only one or two hundredths of a per cent. are present. The reduced copper can also be fused beside a small bead of S. Ph. and tested as in *d*.

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d. A delicate and very characteristic test for copper, often more convenient than *a* or *b*, is made by dissolving *oxidized substances* in S. Ph. on wire in O. F., shaking off the bead into a porcelain dish and treating it in a moderately deep cavity on coal beside a bit of tin, half as large, with R. F., just long enough to fuse the two together. (The tin will, of course, not dissolve in the S. Ph., but only adhere to it.) The bead *when cold* will be distinctly red and opaque, or only slightly reddish, according to the amount of copper present. The red color is due to the formation of cuprous oxide (suboxide, Cu_2O). The R. F. must not deposit soot on the bead. This test is also frequently applied to *alloys* not containing too much lead, tin, or other very fusible metals. In these cases a bead of already melted S. Ph. is placed beside the alloy in a very shallow cavity on coal and the alloy fused in O. F. until enough has been oxidized and dissolved, when the alloy is removed and the glass heated with tin in R. F. as before. Bismuth, § 90, *a*, and antimony, § 96, *b*, interfere with this reaction, although if there is enough copper the test may even then often succeed when the R. F. is continued a little longer, the S. Ph. assuming on cooling rather a brownish-red color. Some judgment will then often be necessary, but a little experience will enable the student surely to identify the copper reaction if it has taken place. Under favorable circumstances the test readily shows one or two-tenths of a per cent. of copper. It will show one-half of one per cent. in otherwise pure lead oxide, but the test *c* is then preferable, since very much lead also darkens the S. Ph. bead in R. F. The presence of large quantities of other strongly coloring oxides interferes with it to some extent, especially those that give deep green beads with S. Ph., like uranium oxide.

e. Sulphides, arsenides and *selenides* should be roasted and tested by one of the foregoing methods, unless they show the copper by *a*, without roasting. Arsenates should also be roasted before reduction when the object of the test is to secure at once reasonably pure copper, since otherwise arsenic remains in part with the copper, rendering it more or less brittle and light colored.

f. 1. *Alloys*, if of suitable nature, may be tested at once as in *d*, or *a*; in filings in the latter case.

Nickel (*German silver, nickel coin*) is tested for copper either by *a*, or by *d*, which is best when testing for more than traces of copper. When *d* is followed, as there directed for *alloys*, the S. Ph. after fusion beside the alloy alone in O. F. will be green when cold if it has dissolved a considerable quantity of the oxides of both metals, because nickel oxide gives yellow and copper oxide a blue

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color; if little copper is present it will be greenish while hot, and with S. Ph. and tin in R. F. will be red.

Brass, if tested with S. Ph., will show only the copper colors, with perhaps a trace of iron. *Gold*, *silver* and *platinum*, are best tested by the flame test for small quantities of copper.

2. *Alloys containing lead, bismuth and little antimony* (for alloys with much antimony, see the end of 2) are treated on ordinary charcoal with boric acid, essentially as directed in § 168, but without observing all of the precautions necessary in quantitative work. It is well to take out the button of metal if it grows very small before showing the color of melted copper and finish the operation beside a fresh bit of boric acid; if only a trace of copper was present the cold boric acid will show a reddish spot of suboxide of copper, where the little button finally disappeared. It is possible also to detect the copper, after separating nearly all of the lead with boric acid, by removing the very small button from the acid and fusing it for some time in O. F. beside S. Ph. on coal. The S. Ph. will either be blue from dissolved copper oxide, or will become red on cooling, after short treatment beside tin in R. F., as described in *d*. If the alloy contained only one or two-tenths of a per cent. of copper, this will finally be left as a minute red button when the operation was finished by means only of boric acid, provided a decigramme or so of the alloy was taken.

The same method of separating lead and bismuth by means of boric acid is used for alloys of gold, whether they contain copper, or nickel, or both. The gold assumes a peculiar greenish hue as soon as the lead is gone, and may then be tested as usual with S. Ph. for nickel or copper, or both, § 78, *a*. When boric acid is used to separate lead from nickel only there will be no such peculiar greenish hue observed in the refined metal, but the nickel will be left as a nearly infusible mass after the lead has been almost entirely removed. This nickel residue is separated from the boric acid and tested beside S. Ph. in O. F. on coal, when it will color the glass yellow. If copper was present with the nickel the glass will be green, even when cold, unless very little copper was present, in which case it may only be green while hot. The presence of the copper can always be confirmed by the red color produced on treating the S. Ph. for a short time in R. F. beside tin on coal.

If much antimony or arsenic is present it should be expelled from the alloy as far as possible by treatment with a strong O. F. alone, after which the alloy is fused with a little test-lead beside borax in R. F. on coal, and the lead finally separated by means of boric acid, as above.

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Note.—It is much better to use boric acid to separate lead, etc., from copper, than to remove the volatile metals simply by prolonged treatment with the O. F. alone, but this can be done when necessary, if not too little copper is present.

3. *Tin* and *zinc* can be tested for copper by oxidizing the fine filings in a gentle O. F. on coal, and then testing the oxides with hydrochloric acid as in *a*. Tin may also be tested by oxidizing it beside a bead of S. Ph. on coal, using a fresh bead when the old one is saturated with tin oxide. After the tin has almost disappeared it is oxidized beside fresh S. Ph., which will either become bluish-green when the residual copper begins to dissolve, or if it then fails to show copper, will become red when treated beside a fresh bit of tin in R. F. The tin oxidizes more easily than the copper, and therefore can thus be separated from it.

A more delicate test can be made by treating the tin as described under the quantitative assay, § 169, *g*.

g. *Arsenides of cobalt and iron,* with or without *nickel,* are first treated on coal with borax in O. F., to detect cobalt and iron, § 76, *d*, and then lead is fused with the remaining button and the whole treated as just described in *f*, 2, to obtain the copper. If there was nickel present it will be found with this copper and can be detected by *f*, 1.

For Metallurgical Products see chapter IV.

Bismuth.

89. As obtained by the reduction assay with soda, § 50, *et seq.*, bismuth appears in globules or shining scales, nearly white, with a very slight reddish tinge, scarcely perceptible on such small surfaces. The globules are imperfectly malleable ; the native, crystalline metal is brittle. It is very fusible, and in a gentle O. F. on coal readily yields a yellow coat, § 37, *b*. The metal is somewhat more easily volatilized than lead, and neither it nor its coat impart any tint to the flame.

The yellow coat of bismuth oxide may, in presence of bismuth sulphide, be accompanied by a considerable white coat of bismuth sulphate, as already stated, § 36, *c*, and this white coat will disappear in part under a strong O. F., leaving a yellow spot of bismuth oxide.

The characteristic tests for bismuth are those in § 90, *a*, *b* and *c*.

90. *a*. In absence of antimony the yellow coat of bismuth oxide may be used as a characteristic test. It is obtained by treating the substance with a good R. F. on coal, and is then scraped off with as little coal dust as possible into a dish, dissolved in a perfectly pure O. F. in S. Ph. on platinum wire, the bead shaken off into a porce-

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lain dish, and fused beside about one-third as much tin, in one solid piece, in a shallow cavity on coal, with a clean R. F., just long enough to melt the two well together. A brownish to perfectly black glass, when cold, will indicate the presence of bismuth *in the absence of antimony*, which does the same. The test is exceedingly delicate, but the student must take care not to blacken the bead by soot from the flame. The test may be made even in presence of a copious yellow coat of oxide of lead, since the S. Ph. bead must be very highly saturated with lead before it will assume even a dark gray tinge with tin on coal. The original substance may often be conveniently used in place of the coat, if soluble in S. Ph.

b. The best test for bismuth, under all circumstances, is that of von Kobell, which is made with the mixture of iodide of potassium and sulphur, just as directed for lead, § 86, *b*. A beautiful red coat of iodide of bismuth forms at quite a distance from the assay, if the test is properly made. Prolonged blowing may also cause a yellow coat of ordinary oxide of bismuth to form much nearer the assay, while at the same time even the purest bismuth oxide will yield at times, simultaneously with the red iodide coat and nearly or quite equal to it in volatility, a yellow coat, which forms generally an interior fringe around the red coat, and seems to be also an iodide of bismuth compound. It interferes in no way with the use of the red coat as a test for bismuth, nor is the absence of the yellow coat to be regarded as showing the absence of bismuth, but it is described here simply to call attention to it and guard against the possibility of its being taken for an indication of lead, which yields also a yellow iodide coat. It is often entirely absent.

Precautions. In presence of much mercury, or cadmium, or sulphides of arsenic or antimony, consult § 86, *b*, *Precautions*. The presence of much lead occasions a yellow iodide of lead coat, as already stated, and this may render the red bismuth coat orange-red or orange, or may entirely conceal it. So also may a very large amount of antimony, by forming a copious white coat. If then the bismuth reaction cannot be obtained by adding more of the iodide mixture to the already heated substance, and again applying the R. F., follow *c*.

c. To detect very little *bismuth* in presence of much *lead* the assay is heated in an open tube, one hundred millim. long and at least eight millim. wide, over the spirit lamp, with somewhat more than its volume of a mixture of one part of iodide of potassium and five of sulphur, when the bismuth sublimate will appear as a dull brick-red band, or sometimes irregular streak, about eight millim. above the lower edge of the copious yellow iodide of lead sublimate. If very much antimony is also present as sulphide it can be sufficiently removed by alternate treatment of the substance on coal in a gentle

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O. F. and R. F. If the antimony is present as oxide it may be necessary to fuse the assay in a deep cavity on coal in R. F. with an equal volume of powdered sulphur, the resulting sulphides being treated as directed just above for sulphide of antimony. In either case the assay, now containing much less antimony, is tested in the open tube with the iodide mixture. As little as two-tenths of one per cent. of bismuth can be detected in otherwise pure oxides of lead and antimony; but delicate and certain as the test is, it must be studied carefully by beginners, or they may easily be misled. The proper way to study it is to begin with lead oxide containing no bismuth, prepared for the purpose if it cannot be purchased, and to compare the sublimate from it with those from two or three varying mixtures of lead and bismuth oxides. In this way the true dull, brick-red bismuth iodide sublimate can be distinguished without difficulty from another orange-colored band, that generally appears near it and consists only of sulphur and iodide of lead, while there can be no danger of being misled by a dark reddish deposit of iodine in the upper part of the tube.

d. Should it be desired to employ the wet way for detecting traces of bismuth in lead compounds, follow § 126, testing the bismuth precipitate so obtained with the iodide mixture as in *b*, rolling both together in the dried filter.

For Metallurgical Products see Chapter IV.

Cadmium.

91. *a.* The characteristic test is the coat, § 38, *a*, and Table VI., obtained by treating the subtance in R. F., or sometimes even in O. F.

When very little cadmium is present with zinc or other metals also yielding coats, a little soda may be added and the cadmium volatilized by a short treatment with R. F., stopping to look for the cadmium coat before the other metals are volatilized.

b. The presence of much *lead* or *bismuth* with *zinc* entirely prevents the recognition of small quantities of cadmium by means of its coat. In such cases, whether dealing with oxides, metals, or alloys, form a coat in R. F., scrape it off, mix it with a little fine charcoal dust, and heat it with a moderate O. F. in a closed tube. Too hot a flame may drive off zinc, forming a zinc oxide sublimate. A reddish-brown ring of cadmium oxide will form a little above the assay, sometimes perceptible only by reflected light; much cadmium would give a metallic mirror, § 31, 2, *h*. This method will detect two-

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tenths of a per cent. of cadmium oxide in any mixture of lead and zinc oxide, and much less cadmium in commercial zinc. *Arsenic*, if possible, must be removed by gently heating the substance in O. F. on coal, since it interferes with the test. This test can be applied in qualitative, wet analysis, to mixed precipitates of copper and cadmium carbonates, or to their sulphides after roasting.

c. Cadmium being reduced with the greatest ease from its carbonate, may sometimes be detected in smithsonite by simple ignition in the closed tube, being reduced by substances in the mineral, as organic matter, iron protoxide, sulphides, etc., and reappearing as a grayish to yellowish-brown sublimate. Carbonate of cadmium serves as a very delicate test for reducing agents, yielding metallic cadmium, or only a film of the oxide, when heated with the substance in a closed tube, according to the quantity and reducing power of the substance.

GOLD.

92. Gold is easily reduced from all its combinations B.B. alone on coal, and, in absence of non-volatile, reducible metals, is finally obtained in gold-colored, malleable, fusible buttons or grains, not perceptibly volatile after long heating. It assumes while melted a peculiar greenish hue, much like that of melted copper. The addition of borax is almost always useful in brightening the surface of the gold and removing traces of oxidizable metals, while a little soda also hastens the elimination of small quantities of arsenic, sulphur, etc. Lead, bismuth, antimony, tellurium, and all volatile bodies can be fully volatilized by a strong O. F. on coal, but are more quickly removed by cupellation, § 83, *b*, which is necessary in presence of copper or other non-volatile metals. If properly performed the cupellation leaves only gold, or its alloys with silver, platinum, and similar noble metals. Silver can be detected by fusing the button beside S. Ph. in O. F. in a very shallow cavity on coal, the oxide of silver yielding an opalescent glass. Gold may be present in large quantity in an alloy with silver and yet not show its yellow color at all. It is then to be separated by means of nitric acid, after previous fusion of the alloy with silver, § 162, *a*. If much platinum, etc., is present, the lead cannot be entirely separated from the noble metals on the cupel, because an infusible alloy results. In this case compare § 93.

Very small quantities of gold are to be detected by an assay, as in § 161, but without especial care in weighing the substance assayed.

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Platinum Group.

IRIDIUM, OSMIUM, PALLADIUM, PLATINUM, RHODIUM and RUTHENIUM belong here.

93. The metals of this group, while differing in many particulars from each other, are usually found together, and are in certain respects so closely allied that they are usually classed together in descriptive chemistry.

Their general behavior, B. B., is given in Table VI.

Plattner gives the following directions for examining certain of their most common natural combinations:

Platinum and *platiniridium*, treated in very fine filings on coal with the glass fluxes, will give reactions for any iron and copper they may contain, although not themselves dissolved. Fused with test lead and borax, they yield alloys which can be cupelled down to an infusible, lustreless, somewhat brittle alloy, still containing lead. On addition of a moderate quantity of gold this alloy can be cupelled nearly, or quite, free from lead, any residue of which is removed by fusion with boric acid, much as directed for copper, § 88, *f*, 2, except that the button of alloy is freely exposed to the oxidizing action of the air, until it shows a bright surface while fused. The alloy thus obtained is hammered thin, annealed by heating on coal and treated with aqua regia. After filtering out any black powder of undissolved iridium, enough ammonium chloride is added to convert all the platinum into ammonium platinic chloride, and the solution is evaporated to dryness over a water bath in which the water does not quite boil. The residue is digested for a short time with alcohol of 80 per cent., after which it is brought on a filter and washed with alcohol until it ceases to color the latter. The gold in the filtrate can be precipitated, after expulsion of the alcohol by heat, with solution of ferrous sulphate, and after fusion to a button with borax can be freed from rhodium, if necessary, as in § 162, *h*. The ammonium platinic chloride on the filter is converted into spongy platinum by careful ignition. For further details of the examination for palladium, etc., see § 162.

All of the above metals are very difficult to fuse; in fact, osmium cannot be fused, but volatilizes at a temperature high enough to fuse even ruthenium.

Finely divided *osmium*, fused in the open tube with potassium nitrate, yields osmium tetroxide fumes of pungent odor and very irritating to the nose and eyes. Heated in the alcohol flame or the

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gas flame osmium powder (platinum residues) renders the flame very luminous.

TIN.

94. *a. Tin* is obtained by the reduction with soda, § 50 *et seq.*, in globules or minute grains, which show, especially when rubbed, a white, metallic lustre. It is malleable and very fusible. In a gentle O. F. on coal it is quickly covered with dull white oxide, but in a good R. F., or just within the point of the inner flame of a strong O. F., it remains bright and slowly volatilizes, forming a coat of white oxide, § 35, *d*, and Table VI.

The characteristic test for tin, apart from the above properties, is that with cobalt solution, § 55, applied either to the oxidized substance or the coat, as the case may be.

b. Oxidized compounds are at once tested on coal in O. F. with the cobalt solution, unless the presence of other bodies renders the formation of a tin oxide coat necessary. This is formed by treating the powdered substance with a very strong R. F. on coal, until a coating of binoxide of tin forms, which is moistened with cobalt solution, after cooling, and ignited in O. F. If little tin is present apply the solution first as in § 79, *b*; and the remarks there made with reference to the presence of cadmium, lead, bismuth and antimony, apply here also.

c. When there is not enough tin present to yield a good reaction, or when much *zinc oxide* is present, the oxides must be finely powdered, reduced on coal with three parts of soda and one of borax (a little charcoal dust is beneficial) in a moderately deep cavity with a powerful R. F., and the resulting reduced globules of tin (perhaps alloyed with other metals) washed out as in § 51. The pure tin can then be recognized by the tests already given, or if alloyed, according to *e*, below, or by the wet way, *f*.

d. Sulphides are first roasted and then treated as in *a*, *b*, or *c*.

e. Alloys are treated on coal with a strong R. F. Alloys of tin with copper yield a tin coat, which can be tested with cobalt solution. Alloys, especially of lead or bismuth with tin, generally become coated with an infusible crust of oxides in a moderate O. F., the formation of which is quite characteristic of tin. By using a very powerful R. F. a coat can usually be obtained, consisting of oxides of lead or bismuth and tin, in which the tin may often be found as directed for zinc, § 79, *d*. If the tin is not thus detected, heat some of the finely divided alloy with a little slightly dilute nitric acid, which dissolves the lead and bismuth and leaves an insoluble, white

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residue of binoxide of tin, which can be collected on a filter, washed and tested as in *a*, *b*, or *c*.

Note.—Antimony must be removed before testing for tin, as directed under zinc, § 79, *d*, Note.

f. Although *tin* can be found in presence of a considerable excess of *zinc* by a reduction from oxidized compounds, which yields metallic tin, yet in presence of much *antimony*, *lead*, or other *reducible metals* yielding coats, a small amount of tin might still escape detection in the reduced alloy. This alloy, or alloys in general containing too little tin to admit of direct detection, B. B., should be treated with nitric acid and tartaric acid, until all the alloy is dissolved, or at least oxidized, the insoluble residue of tin oxide (still containing a little antimony, if that metal was present) filtered out, washed, dried and tested, B. B., on coal, after first removing the greater part of any residual antimony, as in § 79, *d*.

For Metallurgical Products see Chapter IV.

Antimony.

95. The reduction test is not ordinarily used to detect antimony, but it yields, with or without soda, as the case may be, white, metallic globules and grains, which are brittle and very fusible, and in a gentle O. F. on coal rapidly volatilize, yielding a white coat, § 35, *b*, while both the metal and the coat color the inner flame pale yellowish-green. In a moderately strong O. F. antimony volatilizes rather rapidly, and if the assay piece is of any considerable size it continues to oxidize when removed from the flame, becoming covered with acicular crystals of antimonous oxide, especially if gently blown upon with the blowpipe.

The characteristic tests for antimony are the reaction of the coat with S. Ph. and tin, § 96, *b*; the white, rather volatile coat itself, compare § 36, especially *c*, and above all the pale yellowish-green flame, § 96, *a*.

96. *a*. While the presence of *antimony* in *sulphides* is generally quite well shown by the tests in closed and open tubes, §§ 31, 32, the readiest test is afforded by the white coat which nearly all antimony compounds yield on coal in O. F. or R. F., the O. F. being first employed and the R. F. afterward, if necessary to form a coat. This coat will impart to the inner blowpipe flame a very characteristic, pale, yellowish-green color, § 42, 4, *e*, which the student must learn to produce and recognize, since it often affords the only reliable blowpipe test for antimony. It is best obtained by using a small O.

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F. from a gas flame or a clean alcohol flame; the oil flame is equally reliable, but requires more care. The flame coloration is generally too feeble to be readily noticed by beginners in a brightly lighted room, unless the coat is abundant. It is especially marked in the fringing border of the flame as it is deflected back from the surface of the coal. In the absence of substances yielding flames that resemble or mask the antimony flame, this test is conclusive and can be used to test colored coats as well as pure antimony or other white coats. See *g*.

b. The antimony coat can also be tested just as described for bismuth, § 90, *a*, but the student will often fail to detect small quantities of antimony if he does not take care to use only a perfectly pure O. F. to dissolve the coat scraped from the coal in the S. Ph. bead, since the R. F. would reduce the antimony from its oxide and volatilize it. The coat so tested need not necessarily be white, but may have any color due to other metals present. The reduced antimony in the S. Ph. bead does not render it so dark as a corresponding amount of bismuth, but it also may make it grayish-brown to nearly black if abundant. Since bismuth does the same, a test must be made for this metal, § 90, *b*, especially if the substance alone yields a yellow coat on coal in R. F.

c. In presence of *bismuth* the student must depend upon the color of the flame yielded by the more remote and whiter parts of the coat on coal. Should there be so heavy a bismuth or lead coat as to interfere with this test, treat the substance in R. F. on coal beside a little fused boric acid, directing the flame upon the latter flux, which will dissolve the oxides of lead or bismuth, while a nearly pure antimony coat will be formed, which is then tested by the coloration of the flame as in *a*.

d. *Copper* and *tin* alloy readily with *antimony*, so that a little of the latter might fail to yield a coat. In this case fuse the alloy long enough on coal beside a bead of S. Ph. in O. F. to dissolve some of the oxides and then remove the bead of S. Ph. from the alloy and fuse it with tin as in *b*, to obtain the antimony reaction. The presence of bismuth would vitiate this test, and then a larger piece of the alloy must be fused with lead in R. F., and the whole treated beside boric acid as in *c*. Oxides containing copper and a little antimony can be at once dissolved in the S. Ph., unless bismuth is present, in which case they are first reduced to metal and treated with lead and boric acid.

e. As already stated, *antimony* can be detected in any coating not containing *bismuth*, by the test with S. Ph. and tin. Should there be any

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reason to suspect that the reaction is produced by a little bismuth still in the coating, and should the student be unable to obtain a good flame reaction for antimony, the wet way may be employed, submitting to this process some of the coating itself, a very little antimony only being necessary.

f. Galena and other *sulphides* containing much *lead* are best tested for very small quantities of antimony by treating them briefly in R. F. with addition of soda, to prevent the sulphate of lead coat, § 36, *c*, from masking the white antimony coat. Since prolonged treatment will cause a sulphate of soda coat, § 36, *a*, it may be necessary to test as follows: add to the powdered substance, in a rather deep cavity in coal, a bit of iron wire, cover the two with as much powdered borax glass as substance, and with twice as much soda, and treat with a moderate R. F., directed upon the fluxes only, until the lead is reduced to metal. Pour this out on the anvil, cleanse it, if necessary, from slag and iron, by hammering between moist filter paper, and test it with boric acid as in *c*.

g. Tellurium colors the flame green and quite strongly so; it also yields coats, and when both it and *bismuth* are present a little *antimony* might only be detected by the wet way.

h. Arsenic sulphide is tested for a little *antimony sulphide* by heating it *very gently* on coal in O. F., afterward igniting any residue a little more strongly. The exceedingly volatile arsenous oxide coat is then driven off by very gentle heating, when enough antimony oxides will remain on the coal to give a perfectly certain flame reaction, even if only three per cent. of antimony was present and not enough remained to form a visible coat after the arsenous oxide coat was driven off.

Another method consists in gently heating the sulphides in a closed tube, cutting off the lower end, containing now chiefly antimony sulphide and testing this in the open tube.

For Metallurgical Products see Chapter IV.

Arsenic.

97. *a.* Arsenic is a steel-gray, brittle metal, which volatilizes readily without fusing and cannot be obtained in the metallic state by a reduction assay, unless other metals are present with which it can alloy. Arsenic is generally easily detected by heating its compounds on coal in O. F., or in R. F. if oxidized compounds of arsenic are present; they yield a very volatile white coat (often grayish near the assay, § 35, *a*, when much free arsenic is present) and a peculiar garlic (alliaceous) odor, best observed after short treat-

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ment of the substance in R. F. The white coat forms at quite a distance from the assay and disappears when the coal is merely warmed by the O. F., long before the coal begins to glow. Unless too little arsenic is present the assay, or even the coat itself, when quickly heated, also tinges the inner flame light blue. The volatile white coat is a strong indication of arsenic, but some chlorides, as of mercury, § 36, yield coats quite similar, so that in all cases the arsenic odor should also be obtained. With this the student must become acquainted, since it is very characteristic and since arsenic is a constituent of many important minerals. If abundant arsenous oxide fumes and a copious white coat are obtained the student should not breathe the fumes yielded by the substance in R. F. too freely, but when little arsenic is present the odor is best perceived by holding the hot assay close under the nose, after brief treatment in R. F. Too long treatment may volatilize all the arsenic and the student must remember that the odor is not to be obtained from the oxides of arsenic, white coats, etc., by treatment with O. F. alone, but that previous reduction, either by R. F., or by the glowing coal, is necessary to produce it from such compounds.

The addition of soda facilitates the reduction and is generally necessary in the presence of unoxidized sulphur compounds or free sulphur, to prevent the evolution of sulphur dioxide, the fumes of which might conceal the garlic odor. In the absence of selenium, which evolves a strong but different odor, the garlic odor may generally be relied upon to establish the presence or absence of arsenic, but when a little arsenic is combined with nickel or cobalt it may yield no odor even in R. F. In such case treat the compound with lead on coal in O. F., when the garlic odor will be perceptible.

b. The reactions in the closed tube, § 31, and open tube, § 32, are available in many cases. If the fumes escaping from the top of the open tube are passed into the alcohol flame or the flame of a Bunsen burner, they produce a distinct light-blue coloration.

98. *Tests for small quantities of arsenic in doubtful cases.*

a. Metallic arsenides and *sulphides,* or *metals* with little arsenic, are finely divided and fused with six volumes of nitre in a platinum spoon, the mass heated with water, the solution filtered into a test tube, acidified with hydrochloric acid, a little magnesium sulphate in solution added, and then ammonia to strongly alkaline reaction. The whole is boiled a moment, allowed to stand until any precipitate (arsenate of magnesia) that forms has settled, and this is washed by decantation with water containing one-third its volume of ammonia, then transferred to a porcelain capsule, dried, mixed with three

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volumes each of potassium cyanide and *dry* soda, and tested as usual in R. F. on coal to obtain the arsenic odor. It may also be heated in a closed tube, or matrass; at first very gently, to expel any moisture, which must be wiped out with filter paper, and then to low redness. It will yield a sublimate of metallic arsenic, which may be recognized by its mirror-like appearance, § 31, 2, *f*, and farther identified by cutting off the tube just above the sublimate, holding sublimate and tube in the alcohol flame and quickly observing whether any arsenic odor is evolved.

b. Arsenic in small quantities as *arsenous* or *arsenic oxides, arsenites, arsenates* or *sulphides of arsenic* may be detected by the sublimate afforded when the substance is heated with potassium cyanide and soda in a matrass, as directed for the arsenate of magnesia precipitate in *a*. This test is useful also when testing oxide of antimony for arsenic.

c. To obtain a metallic mirror from exceedingly small quantities of *arsenous oxide,* place the assay in a tube drawn out quite fine and sealed at the narrow end, as in Fig. 24, insert above it a splinter of recently ignited charcoal, *c b;* heat this to redness in the alcoholflame and then heat the arsenous oxide. It will sublime, be reduced by the ignited coal and yield a metallic mirror at *d*. The arsenic odor may likewise be obtained by breaking off the narrow end at *c*, holding the sublimate in the flame and quickly trying to observe the odor.

Fig. 24.

d. Arsenates and *arsenites* in which arsenic has not been detected by reduction with potassium cyanide and soda, either on coal or in the matrass, are treated as in *a*, substituting a mixture of equal parts of soda and nitre for the nitre.

This may be necessary when little arsenic is thus combined with reducible oxides, that form fusible alloys with the arsenic.

For Metallurgical Products see Chapter IV.

Molybdenum.

99. *a.* The general behavior of molybdenum oxide is described in Table VI. The reactions with the glass fluxes, § 46, *g*, and Tables II. to VI., especially with borax in R. F., serve for the detection of molybdenum in the absence of non-reducible oxides, and sometimes even in their presence. The coat, § 35, *g*, and Table VI., is also characteristic when pure, the copper-red appearance being especially easy to produce when the coat is as abundant as can easily be ob-

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tained from *molybdenite* and *molybdite*. Molybdic acid in combination with reducible metals can be detected by reduction with borax beside lead on coal, the borax showing the molybdenum reactions after the metals are separated.

b. A little molybdenum can be detected by fusing the powdered substance with three volumes of nitre, if it contains the sulphide, or with three volumes of a mixture of equal parts of nitre and soda, if the oxides, in the platinum spoon, dissolving the mass in hot water, decanting the clear solution, acidifying it slightly with hydrochloric acid, warming it and adding a bit of copper foil, when the liquid will soon become fine dark blue. Tungstic acid gradually causes a feeble bluish color.

c. Molybdic acid can be detected in finely powdered substances by heating them in a porcelain dish with concentrated sulphuric acid and adding alcohol, which causes a fine, azure-blue color, as the mixture cools.

d. Heated on platinum foil with a drop or two of sulphuric acid, until the latter gives off abundant fumes, molybdic acid yields a mass which assumes an ultramarine blue color when breathed upon.

e. Dissolved in S. Ph. in O. F. on platinum wire, the bead powdered and agitated with a strip of zinc and cold dilute hydrochloric acid (one of acid to four of water), molybdic acid yields a green solution, rapidly becoming blue if the bead was highly charged. The blue color remains for some time on pouring off the solution from the zinc, even after being diluted with water and heated, but it rapidly changes to a clear yellowish-brown if at once heated with the zinc. Tin shavings cause the same changes ; see Table VII., § 100, *d*. If the S. Ph. bead is first well reduced alone on coal it yields at once with zinc, or tin, either a brown solution, or a green one, becoming brown on heating, but it never yields a blue solution. The brown color is very dark if much molybdenum is present. If the S. Ph. bead is first reduced thoroughly beside tin on coal, it yields only a faint brown solution, even on boiling with zinc and using a saturated bead. The molybdenum from a very highly charged S. Ph. bead is partly reduced and alloyed with the tin, reappearing as a spongy mass with the reduced tin in the solution, after this has been heated some time.

f. Molybdic oxide dissolved in S. Ph. on wire in O. F., the bead powdered between filter paper, and this paper pressed gently between the folds of a very slightly moistened bit of filter paper, gives a faint reddish, brownish-red, purplish, or blue mass, according to the amount of molybdenum present. This reaction is exceedingly

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delicate and characteristic. The change in color is due, like all the above changes of color, to reduction of the molybdenum trioxide to lower oxides, through the agency of hydrogen when zinc is used, and of organic matter in other cases. A little starch powdered in the agate mortar with the above bead and then slightly moistened, produces a similar reddish to brownish coloration. The test is naturally obscured by the presence of too much cobalt oxide, or other highly coloring oxides, in the bead.

Tungsten.

100. *a.* The general behavior of tungsten compounds is described in Table VI. The reactions with the glass fluxes, § 46, *e*, and Tables II. to VI., especially with S. Ph. in R. F., serve for the detection of tungsten in suitable cases, but there are many cases where the reduction tests in acid solutions, described below, are necessary.

b. Treated according to § 69, *c*, tungstates yield a fine blue color. (Brush's Manual.) The reaction can be used for examining slags, omitting the fusion if the slags can be decomposed in very fine powder by concentrated hydrochloric acid.

c. If tungstates decomposed by hydrochloric acid are finely powdered, heated for some time with hydrochloric acid, the clear liquid poured off and the insoluble yellow residue heated with a little tin and the few drops of acid remaining, it becomes fine blue.

d. Tungstic oxides dissolved in S. Ph. on platinum wire in O. F. and then thoroughly reduced to their lowest state of oxidation in this same bead beside tin on coal, yield, when the bead is afterward powdered and heated with dilute hydrochloric acid (one of acid to four of water) with a few little shavings of tin in a test tube, a beautiful, clear, dark blue solution, so dark as to be almost opaque if much tungsten is present. Poured off from the tin, largely diluted with water and boiled, this solution only slowly loses its blue color. Other oxides give blue colors with hydrochloric acid and tin or zinc under varying circumstances, see §§ 69, 70, 81, 99; but only columbium gives even a tinge of blue color under the above conditions, and it not always. To distinguish with certainty between columbium and tungsten, when necessary, dissolve some of the substance in S. Ph. in O. F. on wire, and at once powder the bead and heat it with the dilute acid and tin shavings, not zinc. Columbates, see Table VII., below, will not produce a blue coloration; tungstates will. Titanic oxides, treated as directed above for tungsten, with S. Ph. in R. F. on coal beside tin, will give a turbid solution, containing a violet precipitate. There is little danger of mistaking this for a

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tungsten reaction; should there be doubt, test as just directed for columbates. Titanium will then fail to give any coloration.

The test just described for tungsten, S. Ph. in R. F. beside tin on coal, is exceedingly delicate. If zinc is used in place of tin in the hot acid solution, the tungsten is rapidly reduced to a still lower state of oxidation than by tin, and yields first a purple, then a reddish-brown solution, the colors being often best seen after dilution with water. The same colors are more slowly produced if the bead is treated only in O. F. on wire before dissolving it.

TABLE VII.—Reduction Color Tests in Solutions.

The oxides are dissolved in S. Ph. on platinum wire, and the bead is then either treated at once, after being powdered, with dilute hydrochloric acid (one of acid to four of water) and tin shavings, or after being first treated in R. F. beside tin on coal. Compare the references given:

THE SALT OF PHOSPHORUS BEAD HAS BEEN TREATED:

OXIDES.	IN O. F. ONLY.	IN O. F. AND THEN IN R. F. BESIDE TIN.
Tungstic Oxide; § 100, *d.*	*Cold*, the solution slowly becomes bluish; *heated*, it quickly becomes blue and at last deep blue and clear, and remains blue for some time when much diluted.	*Cold*, the solution becomes deep blue; *heated*, becomes blue more rapidly, and remains blue when much diluted and boiled, at least for some time.
Molybdenum Trioxide; § 99, *e.*	*Cold*, the solution becomes green, then blue, remaining blue when diluted; *heated*, the solution (undiluted) becomes brownish-yellow or brown.	*Cold*, the solution is brownish-yellow or brown, and may show black particles of the dioxide.
Vanadic Oxide; § 81, *b.*	*Cold*, the solution is green to greenish-blue; *slightly heated*, may become more bluish; *boiled*, bluish-green to green.	The solution is green.
Titanic Oxide; § 69, *d.*	No decided color, but turbid solution, on boiling: with strong acid only is the characteristic color obtained; see reference.	A turbid solution, on heating, from which a violet-colored precipitate finally settles, leaving a nearly or quite colorless solution; the solution may become rose-colored after long standing.
Columbic Oxide; § 70, *b.*	No blue color; turbid solution only (brownish), even on boiling: a characteristic blue color is obtained with zinc; see reference.	As with bead prepared in O. F.; if long boiled the turbid brownish solution may acquire a very faint bluish tinge.

Tantalic oxide gives no colors under any circumstances; *chromic* and *uranic oxides* give the same colors to the solutions which they impart to the beads.

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Tellurium.

101. *a.* Tellurium and metallic tellurides yield tellurous acid in the open tube, § 32, 2, *f*.

b. On coal they afford a tellurium coat, § 35, *e*. To avoid forming the lead or bismuth coats when these metals are present, the powdered substance is treated with fused boric acid on coal in R. F., when a tellurium coat only will appear, unless selenium or antimony is also present, in which case the coat will be less characteristic, and the flame reaction obtained from it may be modified by these substances.

c. If a telluride, or an oxidized compound of tellurium, is mixed with soda and charcoal, melted in a closed tube, and allowed to cool, and a few drops of boiling water added, the solution in the tube will gradually assume a purple color, due to telluride of sodium.

d. Compounds of tellurium gently heated in a closed tube with much concentrated sulphuric acid color it purple or red, the color disappearing with formation of a gray precipitate on addition of water. This test can also be very conveniently made as follows: the substance is treated B. B. on coal, a bit of thin porcelain, as a crucible cover, being placed a little in front of it, the porcelain not lying quite flat on the coal, but so that the fumes evolved by the tellurium compound may pass along the lower surface of the porcelain, between it and the coal. A grayish or brownish film will be formed by the tellurium deposit on the lower surface of the porcelain, if this has not been too highly heated, and when a drop or two of strong sulphuric acid is applied to the film and the porcelain gently heated over the lamp, the acid will assume a fine red color as above.

Selenium.

102. Selenium, in whatever form, can always be detected by the peculiar and offensive odor yielded by free selenium and selenides in the open tube, or by any selenium compound on coal in R. F., soda being then also used if necessary.

Sulphur.

103. *a. Free sulphur* and the higher *sulphides*, like pyrite, readily yield a sulphur sublimate when heated alone in the closed tube, § 31, 2, *a*.

b. Sulphur and *all sulphides* when sufficiently heated with access of air yield sulphur dioxide (sulphurous acid) with its characteristic

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odor. This is very readily perceived in most cases by simply igniting the finely powdered substance in O. F. on coal, and holding the still glowing assay close under the nose, before it has been heated too long. A few sulphides, however, oxidize so slowly that the test must be performed in an open tube, § 32, 1, *a*, when the sulphurous odor will be perceived at the upper end of the tube. *Molybdenite* is a mineral that yields little or no sulphurous odor on coal. In all cases when testing for sulphur in the open tube it is best not to depend only on the odor, but to insert in the upper end of the tube a narrow strip of *slightly* moist blue litmus paper, which will be reddened by the escaping sulphur dioxide.

This test is ordinarily used only to detect free sulphur and metallic sulphides, but fumes of sulphur dioxide and sulphuric acid are yielded by certain sulphites and sulphates of weaker bases, as well as by acid sulphates and sulphites, and these fumes may also redden the litmus paper.

c. A very simple, universal and delicate test for *sulphur*, whether in oxidized or unoxidized compounds, is made by mixing the finely divided substance with two or three volumes of *pure* soda (to which a little borax may be added when it is desirable to keep the soda from sinking into the coal) and treating it in a cavity on coal in R. F. for some time. Sodium sulphide is formed, and on powdering the cold bead, placing it on a bright silver surface and moistening it with a drop or two of water, a more or less distinct yellowish-brown to brownish-black stain of sulphide of silver is produced, which cannot be simply washed off, but may be readily removed by rubbing the silver with a wet bit of charcoal. The fusion should be continued for a sufficient time, enough soda being used to secure a good fusion, and then coal and soda are both cut out and laid on the silver. If selenium, which will have been already detected by its odor, is present, selenide of silver will be formed, producing a similar stain. In such a case the sulphur, according to circumstances, must be detected by *a*, *b*, or *h*. Tellurium compounds may also cause a dark stain on the silver. The flame of coal gas often causes a sulphur reaction ; in such cases an oil, alcohol, or candle flame is used. The borax must be free from sulphur.

d. Usually the appearance of the substance under examination, combined with the tests *b* and *c*, will indicate whether the sulphur is present as *sulphide* or *sulphate*, especially to the practiced operator. In cases of doubt the wet way may be used, or the following test. The finely powdered substance is fused with caustic potassa in a platinum spoon, and this is afterwards placed in a dish with a

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strip of bright silver and covered with water. The silver will soon become brownish if a sulphide was present, but will remain bright with a sulphate, provided no reducing agent was present, like organic matter, carbon, etc.

e. Soluble sulphides dissolved in water will stain bright silver brown, and on addition of hydrochloric acid will evolve sulphuretted hydrogen, known by its odor and by the dark brown color its fumes impart to paper moistened with solution of acetate of lead. Most sulphides evolve this gas when warmed with slightly dilute hydrochloric acid.

f. In the absence of other coloring oxides, *sulphuric* or *sulphurous acid* can be detected by fusing some silica and soda to a clear, *colorless* bead on coal in R. F., and then adding some of the substance and again treating the bead in R. F. A yellow or dark red bead, colored by sulphide of sodium, will result. This reaction will rarely be used, but the fact should be known to avoid the possibility of erroneous conclusions.

g. On fusing a substance containing *sulphur* with soda in R. F., and placing the resulting mass in a watch-glass with a little water and a very small bit of nitro-prusside of sodium, a beautiful purple color is produced. Starch mixed with the soda is better when testing organic compounds, like hair or horn.

h. If it is desired to test for *sulphuric acid* in the wet way, the substance is dissolved in water, or, if necessary, in nitric or hydrochloric acid, the solution, if acid, slightly diluted and barium chloride solution added. A white precipitate, insoluble in boiling hydrochloric acid, will be barium sulphate, indicating the presence of sulphuric acid. Should the substance not be soluble in the acid, it may be well fused on platinum foil with five times its volume of soda, or it may be fused on platinum wire in O. F., after being made into a paste with the soda and a little water. The fused mass is powdered, boiled with a little water, the solution filtered off and slightly acidified with hydrochloric acid. If any precipitate (of gelatinous silica) separates, this is filtered out, and, in any event, barium chloride added as before to the acidified solution. When sulphides are suspected, one volume of potassium nitrate is used with four volumes of sodium carbonate for the fusion. The sulphuric acid in this case will result from oxidation of the sulphur. The potassium nitrate is also added when organic matter is present.

i. The *sodium sulphide* obtained from any sulphur compound, as in *c*, will evolve hydrogen sulphide gas when treated with moderately dilute hydrochloric acid, and *all sulphides* attacked by hydro-

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chloric acid, as *sphalerite, stibnite,* etc., evolve the same gas, which may be recognized by its peculiar odor and by the dark stain of lead sulphide which it produces on filter paper slightly moistened with lead acetate solution and held in the escaping gas.

j. A wet test for sulphur may also be made, according to von Kobell, by mixing a little of the powdered substance with its volume of finely divided iron (*ferrum alcoholisatum* of the apothecaries) and covering the mixture in a short test-tube with a cubic centimetre or so of hydrochloric acid mixed with its volume of water. Strips of filter paper, moistened with lead acetate solution and dried, are suspended in the tube, which is stopped with a cork, and after a minute or two the color of the paper is observed. The tube may occasionally be shaken, but the paper must not be wet by the acid.

CHLORINE.

104. *a.* 1. To detect chlorine in *chlorides,* dissolve a large quantity of copper oxide in S. Ph. on platinum wire in O. F., touch the hot bead to the chloride and then hold the bead within the point of the inner blue cone of flame, when the presence of chlorine will be shown by an intense azure-blue chloride of copper flame. This is perfectly characteristic in the absence of bromides, which cause a similar coloration.

2. A simpler method, equally certain except in presence of large quantities of substances which color the flame very strongly, such as sodium salts, consists merely in mixing the substance on coal with a little copper oxide and a drop of water, and bringing it well within the point of a slightly luminous R. F., when the blue copper chloride flame is produced as before. If the mixture of copper oxide and substance is moistened with a drop of strong sulphuric acid on the coal, instead of water, and the paste first very slightly warmed before a gentle O. F., a very little chlorine will produce an intense blue flame, even in presence of other coloring bodies. This method on coal is especially to be used with substances which might injure the platinum wire.

b. Chlorides soluble in water being added to some small bits of sulphate of iron or copper, lying with a few drops of water on bright silver, will soon stain the silver brown or black. Bromides do the same. The chlorine of insoluble chlorides can be made soluble and thus detected by fusing them with soda on platinum wire in O. F., or on coal if they reduce to metal with soda, as does chloride of silver. Chloride of sodium, soluble in water, is formed.

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c. In presence of *bromine* or of much *iodine*, chlorine can only be detected by the wet way, § 130.

d. All soluble *chlorides* yield with solution of nitrate of silver a white precipitate of chloride of silver, insoluble in nitric acid, easily soluble in ammonia. This readily changes in the light to dark violet. The chlorine of insoluble chlorides is detected after previous fusion with soda, as in *b*.

e. Chlorates deflagrate violently when heated on coal, often almost explosively, and leave a residue of the chloride of the base present, which may be tested by one of the foregoing tests, scraping it off with as little coal dust as possible. In the matrass they fuse, those of the alkalies, alkaline earths and other powerful bases yielding oxygen, the others yielding oxygen and chlorine gases, while all leave chlorides behind.

f. Chloride of silver fused with potassium bisulphate gives a white bead when cold, and this, freed from the potassium salt by heating with water and dried, quickly becomes gray or violet in the sunlight; see also §§ 105, *g*, and 106, *e*.

Bromine.

105. *a. Bromides* treated with oxide of copper, as directed in § 104, *a*, yield a blue flame, very similar to that of chlorides, but having its borders especially tinged with green.

b. Bromides behave like chlorides with sulphate of copper or iron, § 104, *b*.

c. Bromides in solution yield with nitrate of silver yellowish-white bromide of silver, insoluble in nitric acid, rather soluble in ammonia, gradually becoming dark-gray on exposure to light.

d. Bromides fused with potassium bisulphate in a matrass evolve reddish-yellow vapors of bromine, § 30, *c*, 4, best seen by looking down into the matrass held above a white surface. These vapors have a peculiar odor and impart a yellow color to a strip of moistened starch paper inserted in the tube. The presence of iodides obscures the reaction, and bromide of silver yields very little bromine, unless it is first decomposed by fusion with soda, see § 104, *b*, and the bromide of sodium tested as above.

e. Bromates behave like chlorates, § 104, *e*, when heated.

f. In the presence of too much iodine the wet way is necessary for detecting bromine, § 130.

g. Bromide of silver fused with potassium bisulphate becomes dark red while hot, deep yellow on cooling, and on exposure to sun-

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light, § 104, *f*, dark asparagus-green. The more chloride of silver there may be present, the lighter are these colors, the freshly fused and cold bead becoming even pale lemon-yellow, and when exposed to sunlight greenish-gray, if there is more chloride than bromide present.

h. According to Goldschmidt (Plattner, Leipsic, 1878), if a bit of *silver bromide* is placed in an open glass tube, covered with powdered bismuth sulphide and gently heated, yellow bismuth bromide will pass off and form a sublimate in the upper part of the tube. The reaction is characteristic and rather delicate in presence even of silver chloride, which also causes white fumes of bismuth chloride. The presence of iodides tends greatly to obscure the reaction. This test can also be used to detect bromine in various *bromides*. The bismuth sulphide is made by fusing powdered bismuth in R. F. on coal with sulphur, pulverizing the product and again fusing it with an excess of sulphur.

IODINE.

106. *a.* With oxide of copper, § 104, *a*, *iodides* yield an intense green flame.

b. If the test *a* leaves doubt, the *iodide* is fused with potassium bisulphate in a matrass, when violet vapors of free iodine, having a rather chlorine-like odor, are evolved, which color moistened starch paper dark purplish-blue, often nearly black. Iodide of silver gives the reaction best after decomposition with soda, § 104, *b*.

c. If an *iodide* is mixed in fine powder with carbonate of lime and caustic lime (made by strongly igniting the carbonate on coal), the mixture cautiously heated to expel moisture, thoroughly mixed in a mortar with a little mercuric chloride (corrosive sublimate) and heated in a narrow tube, drawn out to a neck a little above the powder, after inserting the latter, iodide of mercury will sublime. It will usually be yellow at first, becoming red afterwards. The drawn out portion of the tube must be cold before the powder is heated and all moisture must be expelled. To apply this very delicate test to residues of water, enough sodium carbonate must be added to decompose all earthy chlorides.

d. Iodides in solution yield with nitrate of silver a yellow precipitate, insoluble in nitric acid, only slightly soluble in ammonia, and but little affected by the light, if pure.

e. Iodide of silver fused with potassium bisulphate in a matrass yields a dark red bead, yellow on cooling and not affected by sun-

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light, § 104, *f*, except in proportion as it contains bromide or chloride of silver. Violet iodine vapors escape during the fusion.

f. Goldschmidt's test for iodine in *silver iodide* and various *iodides*, made in a glass tube with bismuth sulphide, as directed for bromides, § 105, *h*, affords a delicate and characteristic test, in the red sublimate of bismuth iodide which forms a little above the heated assay.

The present writer, however, would recommend, for the detection of larger quantities of iodine on coal, a mixture of equal volumes of sulphur, potassium carbonate and bismuth oxide, all powdered and well mixed. Rather more than one part of this is mixed with the substance to be tested and the whole heated in a gentle R. F., to obtain a bismuth iodide coat; see § 90, *b*.

For detection of iodine in the wet way see § 130.

Fluorine.

107. *a*. Certain minerals containing fluorine and more or less water, yield in the closed tube hydrofluosilicic and hydrofluoric acids, coloring moistened Brazil-wood paper straw-yellow, and depositing a ring of silica inside of the tube, or corroding the glass, near the assay.

b. Fluorine is best detected by mixing the finely divided substance with previously fused and powdered S. Ph. and fusing the whole in an open tube, so that the flame also may enter the tube, having first inserted a strip of moistened Brazil-wood paper in its upper end. In presence of fluorides the paper becomes straw-yellow, and if silica was also present this forms a visible deposit in the tube, which is itself often corroded by hydrofluoric acid; this is evolved especially in the absence of silica and has a peculiar, pungent odor. The corrosion of the tube is best shown by the dull spots on the inside after it has been rinsed out and dried. To avoid fusing the glass it may be necessary to place the mixture on the lower end of a bit of platinum foil, Fig. 25, and insert this partly within the glass tube.

Fig. 25.

c. Fluorides free from too much silicic acid and decomposable by sulphuric acid, like fluorite, cryolite, wavellite, etc., are powdered, mixed to a stiff paste with strong sulphuric acid and gently warmed for some time, in a platinum crucible, or small lead capsule, which is covered with a watch-glass, the lower face of which has been coated with melted wax, through which lines are afterwards drawn with a sharp point of wood, so as to uncover the glass. After half an hour,

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if necessary, the glass is removed from the crucible, and cleansed by rubbing with paper after gently heating it, to melt the wax. The escaping hydrofluoric acid will have etched lines on the glass, where the wax was removed by the wooden point. The lines may be visible only when breathed upon, if little fluorine was present. The heat must not be sufficient to melt the wax while the glass is exposed to the acid.

d. The above test, which is very delicate, can be more simply performed upon minerals containing much fluorine by heating the powdered substance with twice its volume of potassium bisulphate in a closed tube. (Dana.) The heat is continued until the mixture has become nearly quiet and the lower part of the tube dull red-hot. After cooling, the lower end of the tube is broken off by hammering and the upper part of the tube washed out and gently dried, when the inner surface of the tube will be found corroded in cloudy, whitish patches, by the escaping hydrofluoric acid.

CYANOGEN.

108. *a.* Cyanogen is best detected by the wet way. Solutions of cyanides yield with nitrate of silver a white precipitate of cyanide of silver, soluble in ammonia, insoluble in dilute nitric acid. Washed, dried and heated to low redness in a matrass, this is decomposed, leaving metallic silver, which can be dissolved out with nitric acid and will yield a white precipitate of chloride of silver with hydrochloric acid.

b. Froehde's test for *solid cyanides* is a very simple one. A little sodium hyposulphite is freed from water by gentle heating on platinum wire and is then dipped into the cyanide to be tested, after which the two are fused together, until the escaping sulphur begins to burn. Too long heating would decompose the resulting sodium sulphocyanide. The fused mass is next dipped into a few drops of ferric chloride, acidified with a little hydrochloric acid, when a deep, blood-red color will be imparted to the solution. The ferric chloride can easily be made by dissolving a little hematite in hydrochloric acid, or by dissolving iron wire in hydrochloric acid with addition of a drop or two of nitric acid.

For the wet way see also § 130.

PHOSPHORUS (PHOSPHORIC ACID).

109. *a. Phosphoric acid* can often be detected by the flame test,

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§ 42, 4, *d*, in the absence of other substances which give colored flames, especially green; see § 42, 4, *f*, *Note*.

b. Finely powdered phosphates, freed from water by ignition, are placed in a rather narrow glass tube, similar to Fig. 24, § 98, *c*, a slender bit of magnesium wire or ribbon pushed down beneath the surface of the powder, and the narrow part of the tube heated in a somewhat large flame, until the glass fuses. When cold the glass is broken between paper and moistened with a little water, which causes the evolution of phosphoretted hydrogen, having a very characteristic, disagreeable odor.

c. If a small quantity of a phosphate dissolved in nitric acid is poured into twelve or fifteen times as much of a solution of ammonium molybdate in nitric acid in a test tube, a pulverulent, yellow precipitate of ammonium phospho-molybdate will soon form. When very little phosphoric acid is present it may be necessary to wait several hours and to very gently heat the liquid. A mere yellow coloration of the solution does not show phosphoric acid. Arsenic acid alone can cause any error and then only when the liquid has been warmed, but not if the precipitation has been effected in the cold. Substances not attacked in powder by nitric acid may be fused with five times as much of a mixture of three parts of sodium carbonate, one of sodium nitrate, and one of silicic acid, in a platinum spoon, the fused mass boiled with a little water, filtered, boiled with ammonium carbonate, again filtered, and the filtrate tested as above with ammonium molybdate solution.

d. If *copper phosphates* are fused for some time on coal beside an equal volume of lead in O. F., the excess of melted lead and reduced copper poured off from the coal and the remaining slag-like mass treated for some time in R. F., there will be left phosphate of lead, which forms on cooling a glassy bead with irregular crystalline faces. The bead may be slightly yellowish or reddish.

Nitrogen (Nitric Acid).

110. *a*. Nitrates are indicated by their more or less active deflagration when heated on coal, causing the red-hot coal to glow brightly. They are further identified by the yellow vapors liberated on heating the powdered substance mixed with a somewhat larger volume of potassium bisulphate in a matrass. The color is best seen by looking down into the tube held over a white surface.

b. The above test becomes more delicate if the powdered substance mixed with litharge is strongly heated in a rather long tube, in the

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upper end of which is inserted a bit of paper moistened with solution of pure ferrous sulphate, containing a drop or two of sulphuric acid. Any escaping nitrous acid will color the paper yellowish-brown. The paper itself must not become too hot. In the presence of metallic chlorides litharge containing any peroxide of lead would liberate hydrochloric acid or chlorine, which would also color the paper, therefore the litharge must first be tested by heating it with sodium chloride and potassium bisulphate, to see whether it evolves vapors that color the paper.

CARBON.

111. *a. Carbonic acid* is best detected in solids by the effervescence which ensues on pouring dilute nitric acid upon the substance in a test-tube or watch-glass; moderately heating it, if necessary, as well as powdering the substance, should no result otherwise ensue. Carbonic acid gas (or carbon dioxide) being colorless and odorless, is readily distinguished from other gases having characteristic odors, which might be evolved with effervescence, such as sulphuretted hydrogen especially. In case of doubt the gas may be tested by lowering into the tube, nearly to the surface of the liquid, after the copious evolution of gas has ceased, the end of a glass rod to which a drop of clear lime-water is adhering. Carbonate of lime (calcium carbonate) will form and soon render the drop turbid.

b. Free carbon, as diamond, graphite, or coal, ignited in powder on platinum foil, burns more or less rapidly, leaving an amount of ash proportionate to the fixed impurities present, diamond sometimes leaving none. The ash is grayish-white to reddish, according to the amount of oxide of iron in it.

c. Most *organic acids* and their salts, hydrocarbons and organic matters generally, when rapidly heated in the closed tube are decomposed, yielding a black, carbonaceous residue, an empyreumatic odor and frequently an oily distillate.

d. Carbon is detected in minerals except carbonates, metals and alloys, by mixing them in fine powder with two or three volumes of potassium antimonate and fusing the mixture in a matrass in the alcohol flame. Sulphides require six or eight volumes of the antimonate. The matrass, after cooling, is almost filled with water, which is slowly boiled, removed from the lamp, and as soon as it ceases to bubble, treated with a few drops of nitric acid, when the escaping carbon dioxide will cause more or less effervescence. Should not a single bubble of gas be evolved, the substance is free from carbon.

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Silicic Acid (Silica).

112. *a. Silica* (*silicic oxide* or "*acid*") can generally be detected in minerals by its very slight solubility in S. Ph., § 46, *b*. The test is begun by taking up a very small splinter (or a few fine grains, not real *powder*) of the mineral on the S. Ph. bead on platinum wire and treating the bead in O. F. The length of treatment and intensity of the heat should be governed by the ease with which the mineral seems to be attacked by the S. Ph. Usually the bases of silicates will dissolve, leaving a translucent silica skeleton, which retains somewhat the original shape of the splinter, unless the mineral was fused during decomposition. After a little practice the appearance of this skeleton or silica residue is easily recognized.

Some silicates will dissolve entirely, rather easily, and in very considerable quantity. When the mineral does so the silica can, however, generally be identified before it all dissolves and may often be obtained as a permanent skeleton after two or three additions of fresh portions of the mineral, since the S. Ph. ceases to dissolve silica before it ceases to dissolve out the bases; see *Note* at end of *b*.

b. If the mineral is only attacked by the S. Ph. with great difficulty, it becomes necessary to test it in powder, when the silica, if separated from the bases, may remain behind, either gathering into a translucent mass or remaining disseminated in minute translucent specks. The magnifying-glass will often be of service here, by enabling the observer to perceive more closely any evidences of decomposition, as shown by the rounded form of the specks of silica, but the test with powder is often unsatisfactory in examining compounds, although it can be relied upon for identifying pure silica, which is not soluble in it to any great extent, even if in fine powder. For this purpose the size of the S. Ph. bead must be proportionate to the amount of silica to be identified.

Note.—The bead should be examined before cooling, if it becomes cloudy when cold, as it will do if it contains much of any alkaline earth and a few other oxides.

Binoxide of tin and alumina dissolve very slowly, but the former is so easily recognized by other tests that it cannot well mislead the student, while alumina, especially native, as corundum, sapphire and emery, although dissolving slowly, even in fine powder, does yet dissolve to a considerable extent. Native titanic oxide, as rutile, etc., also dissolves rather slowly, but completely in powder. A silica skeleton is translucent; opaque, undissolved residues do not necessarily indicate silica. Certain silicates, especially of zirconia, are scarcely decomposed at all by S. Ph.; silicates of alumina only very slowly even when powdered; yttro-

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tantalite is at first decomposed, with separation of tantalic acid, which afterward slowly dissolves.

c. Since silicic acid is very slightly soluble in S. Ph. small quantities of it, either in fine powder in the free state, as quartz, or mixed with other bodies, may escape detection by the tests above, but it can always be detected by the wet way. The finely powdered substance is mixed with one part of soda and two of borax, wrapped in a soda paper cylinder, and fused in R. F. on coal, § 131, *b*, a little button of silver or gold being added to collect reducible metals, if present. After thorough fusion the metal button, if any is present, is removed, the cold slag finely powdered, heated with hydrochloric acid in a porcelain dish, with occasional stirring (best on a water-bath, § 18, *b*,) until the acid has been evaporated, the residue is gently heated until scarcely any acid odor is perceptible, then moistened with the acid, the dish covered with a watch-glass and gently heated for ten minutes, to dissolve the soluble salts, water is added, and the now insoluble silica is filtered out, washed, dried and tested with S. Ph. as in *b*. In the absence of metals reducible from their compounds by fusion with soda alone, like silver salts, or of metals whose insoluble chlorides or sulphates might cause inconvenience, such as lead, the above test can be simplified by merely fusing the finely-powdered substance, mixed to a paste with five times as much soda and a little water, on platinum wire in O. F., and treating the resulting mass, after powdering it, with hydrochloric acid as above.

If the substance is decomposable by an acid (hydrochloric acid is generally the best, in absence of lead, silver or mercury, in presence of which nitric acid will often serve the purpose) the fusion with soda and borax, or with soda alone, may be omitted and the silica obtained as an insoluble powder simply by evaporation of the acid at first used and subsequent treatment of the residue with more of the acid and then with water as above.

d. Many silicates heated moderately (in *very fine powder* if hard to decompose), with rather more than enough hydrochloric acid to easily cover the powder, the two being well mixed, are decomposed; some forming as the acid slowly evaporates, a perfect, clear jelly, the nature of which is very well seen when stirred with a platinum wire. The test is best made with twenty-five or thirty drops of acid in a watch-glass, over a water-bath. The acid must be added in sufficient quantity, and a little may be added afterward if the mineral seems to be decomposing, but the *gelatinization* will not be observed until the excess of acid has been expelled. If the acid has been too far expelled, so that the residue becomes nearly dry, the

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jelly, even if formed, will lose its gelatinous nature, and if evaporated to dryness cannot again be made gelatinous.

The jelly is silica, separated in a hydrous state, and the phenomenon of gelatinization, being a very important means of distinguishing silicates, should be carefully studied. Some silicates, as calamine [11], gelatinize with great ease; others, as wollastonite [65], only after being treated with the acid in the finest powder for fifteen or twenty minutes.

Other silicates [40] are decomposed by hydrochloric acid, but yield only slimy, flocculent, or pulverulent silica, instead of a stiff jelly. Others are attacked after strong ignition, while still others are scarcely or not at all affected [46]. A proper acquaintance with the phenomena attending the decomposition of silicates by acids is only to be acquired by careful experiments.

e. In oxides containing no free acids not volatilized B.B., as phosphoric, boric, titanic acids, etc., silica is indicated by the effervescence which ensues on fusing the ignited substance in powder with dry soda on coal. Compare § 54, *a.* The test can only be regarded as of negative value.

Boron (Boric Acid).

113. *a.* The general characteristic test for *boric* (*boracic*) acid is the green color, with a slight yellowish tinge, which it imparts to the flame, § 42, 4, *c.* Free boric acid, certain borates, like *boracite*, and silicates, as *datolite* and *axinite*, show this reaction when treated alone; other borates, as *borax* and *ulexite*, containing soda, very transiently after being moistened with strong sulphuric acid; while other silicates, as *tourmaline* [66], although containing considerable boric acid, must be finely powdered, mixed to a paste with a drop of water and some of Turner's flux and the paste heated on platinum wire, just within the inner flame, at first gently, then more intensely. A very transient yellowish-green flame, usually requiring close observation, will be visible just as the assay fuses, even when very little boric acid is present. In ordinary cases, as with tourmaline, one volume of the flux and one of the substance are mixed, but in doubtful cases three or four times as much of the flux should be used.

b. Borates in powder, if mixed with sulphuric acid to a paste, and then gently warmed with a little alcohol, will yield a green flame, when the alcohol is kindled, especially if the alcohol is extinguished after burning a little while and then relighted. Metallic chlorides may interfere with this test, since they can sometimes cause a green flame alone.

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c. Iles proposes to mix the powdered borate with glycerine, either at once, or after it has been calcined, moistened with sulphuric acid, and ignited to expel the acid, and to burn the paste on platinum wire in the alcohol, gas or blowpipe flame, when the green coloration will appear. The result is very distinct, but none of the tests are so generally applicable as Turner's, which fails only in presence of other substances that might color the flame green.

d. 1. *When there is doubt whether the green flame may not be due to copper, etc.*, the substance should be dissolved in water, hydrochloric acid added to slightly acid reaction and turmeric paper partly dipped in the solution for a time, after which the paper is dried at a temperature not above that of boiling water. Boric acid colors the paper a characteristic red. Turmeric paper becomes blackish-brown if moistened with too strong hydrochloric acid, especially after drying; alkaline solutions turn it brown to deep brownish-red; hydrochloric acid solutions of zirconium, molybdenum and ferric chloride also give it a reddish-brown, orange-red or brown color, and various other solutions affect turmeric paper, but do not produce a color which will mislead the experienced operator. Sodium carbonate solution turns the red color produced by boric acid to a dark blackish-green, but dilute hydrochloric acid restores the red color. Compare § 67, *b*.

2. Substances insoluble in water should be decomposed by hydrochloric acid and the solution diluted with water. If not decomposed by hydrochloric acid the substance should be fused (on platinum wire if suitable), after being mixed to a paste with about four volumes of soda and a little water. After three or four beads have been thus made they are powdered, boiled with a *little* water, the solution filtered, slightly acidified with hydrochloric acid and tested with turmeric paper.

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CHAPTER IV.

SYSTEMATIC BLOWPIPE EXAMINATION OF SUBSTANCES.

114. No method of blowpipe analysis can be devised like the systematic methods of the wet way, which admit of the successive isolation and identification of all the different constituents of inorganic compounds. A very large number, however, of the metals can be systematically tested for, in very complex mixtures, while special tests will serve to detect a still larger number, together with many of the non-metallic elements and their compounds. A thorough acquaintance with the resources of blowpipe analysis will enable the chemist to apply these tests with advantage, while directions will be given in the following pages for systematic work covering nearly all the cases in smelting operations.

I. Preliminary Examination.

When it is desired to learn as much as possible regarding the composition of an unknown substance, both for the purpose of learning what constituents may or may not be present and of identifying such as may be unaccompanied by other similar ones, that would prevent their identification by such simple tests, the examination is best made in the following order. If the reaction is not decisive it will always afford indications as to the desirability of applying the special tests, to be found in Chapter III., under each element.

The examination is made :—

1. In the *closed tube*, § 29.
2. In the *open tube*, § 32.
3. On *charcoal alone*, § 33.
4. On *charcoal with soda*, § 50.
5. With the *glass fluxes*, on wire or on coal, according to the nature of the substance, § 44.
6. More limited in their scope are the following tests :

a. Flame-coloration (and fusibility). §§ 42, 40.

b. With potassium bisulphate (see nitric acid, chlorine, bromine, iodine, fluorine).

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c. Reduction color tests (see titanium, columbium, tungsten, vanadium, molybdenum).

d. With potassium nitrate (see osmium and manganese).

II. Special Schemes for Complex Substances.

115. It is not worth while for the student to undertake the examination of very complex substances for all their constituents, since this cannot be done. It is better to learn how to apply special tests for the substances that can be found with the blowpipe under all circumstances and to resort to the wet way for the others, Chapter V.

Very much may, however, be done in the way of systematic analysis of the compounds most frequently met with in metallurgical operations, and the scheme which follows is intended for this purpose, as well as for the detection of such substances in general as admit of detection, even in complex mixtures.

The excellent scheme arranged by Prof. Egleston and published by him in the *American Chemist*, April, 1872, p. 383, is here given, with explanatory notes and some alterations by the present writer, together with an introductory note.*

Introductory Note.—The scheme is to be varied according to circumstances.

a. Oxides and substances containing much sulphur, with or without arsenic, are best tested by the scheme, p. 150, directly, unless their nature is known, as in case of metallurgical products, when the special directions, §§ 116 to 124, will serve best, and they should be followed, if applicable, whenever the nature of the substance under examination is sufficiently known, whether it be a sulphide or not.

b. Alloys free from arsenic can be recognized by their metallic lustre, almost universal malleability (except some of those containing antimony and tin, which metals are known in general by their coats) metallic appearance of their powder when they are brittle, and freedom from sulphur (except traces). Such alloys are first subjected to the tests under **A**, p. 151, after which it will generally be best to treat them by special methods, according to their nature.

If they are *infusible* treat them beside borax on coal in O. F., until the borax has taken up any oxidizable metals in sufficient quantity, and then treat this borax according to the Scheme, p. 153, **B**,

* Prof. Egleston's scheme is given in a concise form on p. 160, as it appeared in the author's translation of Plattner, 2d Ed., 1873, with the author's note. This form may be more acceptable to those accustomed to its use.

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2, *b*, *et seq.*; or else alloy them with lead and treat them as immediately follows here.

If they are *fusible* classify them, if possible, as follows :—

Alloys showing much *tin*, § 94, *e*, and a considerable quantity of *lead*; follow § 120.

Alloys consisting very largely of *lead* with other *volatile metals;* they will, after long heating alone with the point of the inner flame on coal, leave very little residue ; follow raw lead, § 119.

Alloys containing perhaps some *lead, but leaving a considerable residue,* which with lead and boric acid, §§ 88, *f*, 2 ; 78, *c*, proves to be *copper* or *nickel;* follow German silver, § 121, and bronze § 122, according to the constituents sought and according as much or little *tin* is present.

c. Alloys containing arsenic (having also metallic lustre, but often brittle) are classified, if possible, as follows :—

Alloys with *much lead* and leaving, when treated alone on coal with the point of the inner flame, *very little residue;* follow raw lead, § 119.

Alloys *leaving a considerable residue:* the residue is tested as for speisses, § 116, if a preliminary test with borax shows considerable *cobalt* or *iron*, § 76, *d;* otherwise as for black copper, § 118, especially if a test with lead and boric acid, § 88, *f*, 2, shows the presence of considerable *copper*.

There are few alloys met with in smelting operations or in commerce which cannot be brought under the above classes ; such are to be tested as directed in the scheme, which serves also for metals not provided for in the special directions cited. (Alloys containing *aluminium* are to be tested for this metal by the wet way, but any copper, zinc or traces of iron which they may contain can be found as under German silver, § 121. *Platinum* and similar metals must be finally tested for in the wet way also).

SCHEME FOR COMPLEX SUBSTANCES.

The substance may contain **As—Sb—S—Se—Te—Fe—Mn—Co—Zn—Sn—Ni—Cu—Ag—Au—Pb—Bi—Cd—Cl—Br—I—CO_2—SiO_2—N_2O_5—SO_3—P_2O_5—H_2O.***

The detection of small quantities of **Sb** in presence of both **Te** and **Bi**; of very little **Pb** in presence of **Bi**; of **Fe** in presence of both **Co** and **Mn**; of **Zn** in presence of **Sn**, and of **Cl** and **Br** in presence of one another or of **I**, is not always certain.

* For **W, V, Cb, Mo, U, Cr, Ti**, see **D.**

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A. Examination for volatile substances yielding odors or coats on charcoal, § 33, et seq.

Treat the substance alone on coal in O. F., followed by a *gentle* R. F., especially to find **As—S—Se—Te—Sb—Pb—Bi—Cd.*** (**Zn, Sn** and other bodies may yield coats; their detection will be given under **C**. Compare also § 36).

1. **Se** and **As** will be known by their odor; **Te** by the *bright* green color which any coat containing it will impart to the inner flame (bluish-green when **Se** is also present. Copper chloride yields an intense azure-blue flame; its iodide a green flame; its bromide a blue flame, inclining to greenish). Confirm **Te** by **C**, 2, *j*.

2. If a coat forms it calls for the following tests also.

a. For **Sb** by the pale yellowish-green color imparted to the flame by various parts of the coat, especially the white parts, § 96, *a*; confirmed, in absence of **Bi** by the test with S. Ph. and tin, § 96, *b*.†

b. If a yellow coat (cold) is obtained, with soda if not without, test a fresh portion of the substance with potassium iodide and sulphur to identify **Bi**, § 90, *b*, and **Pb**, § 86, *b*.‡

c. **Cd** is known by its characteristic coat, § 91, *a*; if necessary, confirm the presence of **Cd** by **C**, 2, *k*.

d. **As** usually gives a white coat, *exceedingly volatile*, § 97, *a*, but the coat alone is not a sure indication of **As**; certain volatile salts give a similar coat. Confirm **As** by **C**, 2, *a*.

B. Examination for metals whose oxides are not reducible from borax alone, Fe—Co—Mn; also for the reducible metals Ni—Cu—Ag—Au.

Test in the open tube for **S, As, Sb** and **Se**, § 32, and if any of these are found, or if any considerable quantity of **As** has been found already on coal, thoroughly roast a sufficient quantity of the substance on coal, § 43, until it no longer gives fumes of **S, As**, or **Se**, and proceed as below, 1, 2. If no reactions for **S, As, Se** or **Sb** are obtained in the open tube, nor for **As** on coal, and the substance is brittle, yielding a non-metallic, lustreless powder, the roasting is

* If no yellow coat forms it is always well to test a fresh portion of the substance with soda in R. F. for a colored coat, and this is necessary in case volatile chlorides, iodides, etc., are present.

† To detect a little **Sb** more surely in presence of **Bi**, reduce with soda and test the alloy by § 96, *c*.

‡ Tube tests will show small quantities of **Bi**, § 90, *c*, and **Pb**, § 86, *c*.

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omitted and the powder tested as below, 1, 2. (If the roasted substance is entirely or largely reduced to metal, treat it as an alloy).*

1. Test some of the roasted substance, if convenient, for **Cu** with hydrochloric acid, § 88, *a*, confirming the presence of **Cu** afterward as in 2, *b*, below.

2. *a*. Dissolve some of the powder in borax in O. F. on platinum wire, observing any colors imparted to the bead by gradually increasing quantities, since they may be characteristic.†

(If no color is observed, even in the hot bead, pass at once to **C**; if the somewhat highly charged, hot bead is yellowish, but the cold bead colorless, only a very little **Fe**, or the rarer oxides in **D**, can be present, as far as regards the substances to be sought for especially here.)

If the cold bead is colored at all, saturate the bead highly, shake it into a porcelain dish § 44, 1, *c*, and repeat the operation once or twice, as may be judged necessary to secure a sufficient quantity of any reducible metals in it.‡

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* A general method for alloys, but not so convenient for special cases admitting of classification according to the introductory note, p. 149, is as follows: Treat the alloy alone on coal, in a strong R. F., to find (**As**, **Sb**, scarcely perceptible in roasted substances in general) **Pb**, **Bi**, **Cd**, **Sn**, **Zn**, confirming each by the tests given in **A** and **C**. After it has ceased to yield any coat or arsenic odor test a small portion of any residue for **Cu**, with hydrochloric acid, § 88, *a*, and treat the rest beside a piece of borax glass in O. F. on coal (noting, if possible, any successive indications of **Fe**, **Co** and **Ni**, § 76, *d*, especially when arsenic is present in any quantity). After treating the residue for some minutes, stopping sooner, however, if it all dissolves, and changing the borax as often as it becomes opaque from dissolved oxides, treat all the borax as in 2, *b* (with a gold button if no **Cu** was found; otherwise lead may be used) to find **Fe**, **Co** and **Mn**. Then treat the gold (or lead) button for **Ni**, **Cu** (and **Ag**) as in 2, *b*, *b*. If there was any of the original residue which did not dissolve in the borax, this may be alloyed with the gold button, with addition of a little lead, by fusion beside borax in R. F. (or simply with the first lead button, if no gold was used) and the **Ni** and **Cu** found as before.

† If the roasted powder contains *any metallic particles* it is not safe to test it on wire, but it should be well mixed with sufficient powdered borax glass and fused with this in R. F. on coal to a single bead (possibly with some reduced metal adhering to it), after which the reduction is completed as in *b*.

‡ Unless the test with hydrochloric acid has already shown **Cu**, it is often worth while to make a reduction test of the roasted powder (or the raw substance if no roasting was found necessary) with soda and borax, since if no metallic buttons, scales, or magnetic powder (**Ni**) are thus found, even after washing in the mortar, very little of any reducible metal can be present, and unless very small quantities of such are to be sought for it will only be necessary to look for **Fe**, **Co** and **Mn**. In such a case simply test the powder with borax (and with S. Ph.) on wire in O. F. and R. F., and on coal with tin in R. F., referring to Tables I. to VI., and §§ 72, 74, 76, for the necessary explanations of the cause of any mixed colors produced. S. Ph. may be useful to detect **Fe** in presence of **Mn**, § 74, *d*.

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b. Treat these beads with a strong R. F. on coal beside a bit of test lead, about half as large as an ordinary borax bead. If previous tests have shown the presence of considerable **Pb** or **Bi** no test lead need be used. (If no **Cu** has yet been found it is better to employ, in place of the test lead, a gold button weighing fifty to eighty milligrammes, to collect the reduced **Ni**, especially when but little of the latter is likely to be present, § 78, *c*, 1, 2, and to use the gold, in absence of **Cu**, even if **Pb** or **Bi** are known to be present.)

The borax beads must all collect into one bead and the lead (or gold) button must gather up all the reduced metals. The operation should be continued long enough to ensure the reduction of all reducible metals, and if the borax spreads over the coal it must be brought into one bead by a continued and powerful R. F., § 44, 1, *d*; otherwise **Cu** or **Ni** may remain in it and give deceptive colors afterwards. When the reduction is complete the glass is drawn out, to observe its color, and when cold is separated from the button by hammering between paper. The glass is kept for test *a*, the button for *b*.

a. Non-reducible oxides, of **Fe, Mn, Co,** will remain in the borax glass. (If this gives too dark colors dilute it by adding bits of pure borax glass.) If it is colorless treat a portion of it on wire in O. F.; an amethyst color will then show **Mn.** If it is blue and remains so in O. F. on wire, both when hot and cold, it contains **Co.** If it is pale vitriol-green to dark bottle-green and in O. F. on wire becomes yellow to dark red while hot, lighter yellow on cooling, it contains **Fe** (with very little **Fe** it may be colorless in R. F. and yellow only while hot in O. F.). For mixed colors of **Co, Fe** and **Mn** see references in the last foot-note, p. 152; (for **Cr, Ti, W**, etc., see **D.**)

b. Reducible metals, **Ni, Cu, Ag, Au** will remain in the button, as will also **Pb, Bi, Sn,** etc., except in so far as these last may partly volatilize.

The method to be next adopted will depend on the conditions under which the reduction in *b* has been performed.

If no **Bi, Sb** or **Pb** was present, and a gold button was used, treat the button at once, unless it contains reduced **Sn,** see the second foot-note p. 154, with S. Ph., § 78, *a*, to detect **Ni** and **Cu.**

If **Bi** or **Sb** was present, with or without **Pb,** and a gold button was used, treat the button alone on coal with the point of the inner flame until most of the **Bi** and **Sb** have been removed, alloy the residue with a little lead, by fusion beside borax in R. F., and finish

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with boric acid, etc., according to the last few words of § 78, *c*, 2.* If **Pb** only was present and a gold button was used, the **Pb** must also first be removed with boric acid.

If test-lead was used in place of a gold button, or if no gold button was used, and the substance originally contained lead, separate the lead with boric acid, § 88, *f*, 2, and test the residual button for **Ni** and **Cu** just as directed for the nickel-bearing gold button, § 78, *a*, but of course without adding any gold.†

In all cases of doubt confirm **Cu** in the S. Ph. bead by § 88, *d*.

(Any **Ag** or **Au** in the substance will be found by cupelling the residual button from the treatment of the lead with boric acid; if a gold button was used to collect **Ni** and **Cu** it will contain the **Ag**, but a special test must be made for the **Au**. See also **C**, 2, *e*.)

C. Special and Confirmatory Tests.

1. *Tests to be applied to the roasted substance, or to the raw substance if roasting has not been found necessary.*

a. **Zn** and **Sn**‡ Expel volatile substances (**Cd, Pb, Sb, Bi**) by gentle O. F. on coal, then reduce strongly with soda and borax (and a little charcoal dust when **Sn** is sought for) in a moderately deep cavity on coal, shaved away somewhat on the further side so as to allow the formation of a coat, § 50; test any coat with cobalt solution for **Zn**, § 79, and for **Sn**, § 94. Also wash the powdered slag and test any metallic residue for **Sn**, § 94, *a*, *e*.

b. **Mn**. Test with S. Ph. (or soda) and potassium nitrate, § 72, *b*, *c*.

c. **Si**. Test with S. Ph., § 112, *a*.

2. *Tests to be applied to the raw substance.*

a. **As**. Test with soda in R. F. on coal, to get the odor; or make suitable special tests, § 98.

b. **Sb**. If the substance contains no **Bi** dissolve a little of it in S. Ph. in O. F. on wire and test with tin, § 96, *b*; if it cannot be tested

* The **Bi** and **Sb** can also be removed from the gold button by prolonged treatment with the point of the inner flame alone, but the method is tedious and less suitable for the detection of traces of **Cu**; it may be adopted with advantage, however, when *very little* **Ni** is to be sought for.

† If the button from the reduction with borax contains *very much* **Sn** it is best to remove this to a great extent with S. Ph., § 88, *f*, 3, and to alloy the residue with a little test-lead, by fusion beside borax in R. F., after which the boric acid is used as above.

‡ If chlorides, iodides, or other very volatile compounds of **Zn** or **Sn** are suspected, the preliminary treatment with O. F. must be omitted. Potassium cyanide reduces tin chloride better than soda.

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on wire, on account of its metallic nature, treat a little of it on coal beside an already formed S. Ph. bead, § 96, *d*. If alloyed with **Cu** or **Sn** proceed also as in § 96, *d*.

c. **S**. In absence of **Se** or **Te** test with **soda**, § 103, *c*; in presence of **Se** test in the open tube, § 103, *b*, (but oxidized sulphur compounds will generally yield no reaction then.) To distinguish between sulphides and sulphates (or sulphites) follow § 103, *d*.

d. **Hg**. Follow § 84.

e. **Ag** and **Au**. Follow §§ 83, *b*, and 92; test the button from cupellation for **Au** by § 162, *a*, using ordinary strong nitric acid. If it has a yellow color test it for **Ag** by § 92.

f. **Cl**, **Br** and **I**. Test with oxide of copper §§ 104, 105, 106; also with potassium bisulphate, to distinguish between **Cl** and **Br**, § 105, *d*.

g. H_2O; § 30, *d*.

h. CO_2; § 111, *a*.

i. N_2O_5; § 110.

j. **Te**; § 101, *d*.

k. **Cd**; § 91, *b*.

l. P_2O_5; § 109, *b*.

D. Detection of the rarer metals, Cr—Cb—Ti—U—V—Mo—W.

When these occur in sufficient quantity to be of interest tests made with both borax and S. Ph., on the oxidized substances, will afford characteristic colors at once, Table I. (with the exception of **Cb**, see Table VI.), or colors indicating their possible presence. Those which are liable to occur together will be mentioned below. If iron or other coloring oxides interfere with the borax and S. Ph. tests make special tests as below.

Cr; § 68, *b*: **Cb**, § 70, *b*; in presence of **W**, § 70, *c*: **Ti**; § 69, *d*, *c* or *b*: **U**, § 80, *b*: **V**, § 81, *a*: **Mo**; § 99, *d*, *e*, *f*: **W**, § 100, *d*; in presence of **Cb**, § 70, *c*.

III. Examination of Metallurgical Products and Paints.

Speisses (see § 76, d).

116. The various speisses contain *arsenides of nickel, cobalt* and *iron*, in varying amounts, with more or less *copper, lead, bismuth, antimony, zinc* and *sulphur*, according to the nature of the ores from which they are made and the processes they have undergone. *Silver* and *gold* are often present in speisses from certain mixed ores.

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1. Powdered and tested in the open tube with blue litmus paper, they may show both *sulphur* and *arsenic*, § 32.

2. A small fragment is heated alone in a shallow cavity on coal in R. F., until it has nearly ceased to yield arsenical and other fumes, after which a bit of borax glass is placed beside it and the whole treated with the R. F. (If the speiss, owing to the presence of much iron, is rapidly covered with a dark crust and does not readily yield fumes a little borax should be added at once, and the speiss afterwards treated alone, when most of the iron has been dissolved.)

Any considerable quantity of *lead* or *bismuth* will now be shown by a yellow coat, which may be accompanied by a white coat of lead sulphate (in case much sulphur is present) while oxide of *antimony* may also be present in the white coat, and can be detected by the color which the coat imparts to the flame, § 96, *a*. When very little antimony is present it can be detected by the test, § 96, *b*, unless a special test on some of the fresh powdered speiss shows the presence of bismuth, in which case follow § 96, *d*. When much lead sulphate is formed it is well to add soda to some of the fresh powdered speiss and treat this on coal in R. F., when the sulphur will be mostly retained as sodium sulphide and purer lead oxide and antimony oxide coats formed.

The special test made for bismuth with potassium iodide and sulphur will also serve to detect lead, if this is not in too small proportion as regards the bismuth, while in absence of bismuth the merest trace of lead will be shown by it, §§ 90, 86.

Even in presence of a moderate amount of antimony, any *zinc* present can be found in the coat obtained with soda, as above, by the proper use of cobalt solution, § 79.

3. After expelling most of the volatile constituents from the speiss beside borax in R. F., *iron* (which will also already have been dissolved by the borax in part), *cobalt*, *nickel* and *copper* can be detected as in § 76, *d*.

4. *Silver* and *gold* will require a special test by cupellation, §§ 83, *b*; 92, a little of the speiss being melted in R. F., with enough lead beside borax on coal, until well fused with it, when the lead is refined and cupelled.

Speisses containing very much *sulphur* should be roasted and treated like matts, § 117.

Matts.

117. These contain mainly *sulphides of iron, copper* and *lead*, with more or less *zinc, cobalt, nickel, bismuth, antimony* and *arsenic*, according to the nature of the ores and the processes of smelting.

1. *Sulphur* is found by the open tube test with litmus paper, § 32, and *arsenic*, in any great quantity, by its odor alone or with soda.

2. Fused alone on coal in R. F., or with addition of soda, if necessary, the matt will yield indications of any *lead, bismuth, antimony, arsenic* or *zinc* present, by appropriate tests; compare speisses, § 116.

3. The well roasted matt is next treated according to the scheme, **B**, p. 151, for *iron, cobalt, nickel* and *copper*.

Silver and *gold* are found by a rough assay, §§ 83, *b*; 92.

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Silica, derived from disseminated slag, may often be detected in the roasted matt by means of S. Ph., § 112, *a*.

Black Copper.

118. *Copper, lead, bismuth, tin, zinc, antimony, arsenic, sulphur, iron, cobalt* and *nickel* may be present in this substance, the method for which is also applicable to several somewhat similar products of smelting processes.

1. Filings or powder are tested for *sulphur* in the open tube with litmus paper.

2. *Arsenic, lead, antimony, zinc* and *bismuth* are in general to be tested for as under speisses, § 116, but the more delicate tests may have to be made for arsenic and tin, which can often be found only by the wet way.

3. To detect *iron* and *cobalt* a bit of the black copper is fused in a shallow cavity on coal in O. F. beside borax glass for a short time, the metal removed and the glass bead treated in a deeper hole with R. F. beside a bit of lead. Iron and cobalt will remain in the borax bead ; compare scheme, p. 153, **B**, 2, *b*, while copper and other metals, previously oxidized and dissolved in the borax, will be reduced and will pass into the lead.

4. To detect *nickel* the lead from 3 is melted together with the copper there used in R. F. beside borax, until the two are well alloyed, when the lead is removed with boric acid, § 88, *f*, 2, and the resulting copper button tested for nickel, § 78, *b*.

5. *Silver* or *gold* are found by a special assay, §§ 83, *b* ; 92.

Raw Lead.

119. It may contain *sulphur, arsenic, antimony, bismuth, copper, zinc, iron, silver*.

1. *Sulphur* is detected by the test with soda, § 103, *c* ; *arsenic*, if present, evolving its characteristic odor during the fusion ; bismuth, § 90, *b*, *c*.

2. *Antimony* is best detected by § 96, *c* ; the same portion of lead serving further for the detection of *copper*, § 88, *f*, 2.

3. *Zinc*, unless in rather small quantity, can be detected by § 79, *d*.

4. *Iron* is found by fusing some of the lead beside a little borax in a shallow cavity on coal, directing the R. F. only on the borax for some time, but allowing air to act on the exposed lead. Afterward the lead and borax are removed to a deeper cavity, the borax covered with a good R. F., which at the same time, with the aid of the coal, protects the lead from oxidation, so that all reducible metals may be reduced and taken up by the lead, leaving nothing but iron in the borax. The borax is removed and fused on wire in O. F., when any iron present will give a yellow color, at least while hot.

5. *Silver* and *gold* are found by cupelling some of the lead, after first refining it in O. F. on coal beside borax, § 159, *c*, or else by testing the copper, if any, from 2, according to § 159, *b*.

Impure Tin and Alloys with much Tin.

120. 1. *Sulphur* is found by § 103, *c*.

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2. *Lead* by § 86, *b* or *c*; *bismuth* by § 90, *b* or *c*.

3. *Iron* by fusing beside borax on coal, directing the inner flame on the borax, which is afterward treated in O. F. on wire to get the yellow iron color; see also § 119, 4.

4. *Copper* by § 88, *a*, or *f*, 3.

5. *Tungsten* (very rarely in any quantity) is found by oxidizing some tin beside S. Ph. on coal, directing the O. F. upon the tin. After saturating several beads they are fused together, with a large R. F. in a deep cavity in coal, beside a small bit of the tin for some time, when the bead, with the tin adhering is treated with hydrochloric acid, to get the blue solution showing tungsten, § 100, *d*.

6. *Antimony* by § 96, *d*; *arsenic* by the odor in R. F., or by § 98, *a*, if very little arsenic is to be sought for.

German Silver.

121. It contains *copper*, *nickel* and *zinc*, with more or less *iron* and *cobalt*.

1. Alone on coal in R. F. gives a *zinc* coat; confirm with cobalt solution.

2. The same button is afterward oxidized beside borax for some time, the metal removed and the borax treated with a strong R. F. in a deep cavity on coal, beside a bit of lead, which takes up all the *nickel* and *copper*, leaving any *cobalt* and *iron* in the bead. The glass bead will now be blue if containing *cobalt* alone, or *cobalt* with very little *iron*. (A similar alloy with considerable iron would render the bead greenish-blue.) If the bead is now oxidized on wire it will, at least while hot, appear green or greenish-blue in presence of both *iron* and *cobalt*, but yellow if *iron* alone was present.

3. *Copper* and *nickel* are found by § 88, *f*, 1.

Bronze, Bell-metal, Gun-metal.

122. The last two generally contain *copper* and *tin*, with a little *iron* as an impurity; the former may contain also *zinc* and *lead*.

1. The *copper* is found by oxidizing the metal beside S. Ph. on coal and then testing the bead with tin, § 88, *d*.

2. The *tin* is found by fusing the metal in R. F. on coal and testing the coat, § 94.

3. The *lead* is best found, if in small quantity, by means of potassium iodide and sulphur, § 86, *b*, *c*.

4. The *iron* is found as directed under German silver, § 121.

5. The *zinc*, unless in quite small quantity, can be found by fusing the metal alone on coal in R. F., until a slight coat forms, which is tested with cobalt solution, previously spread on the coal and gently dried. Zinc is much more volatile than tin and will form its characteristic coat first.

6. *Bismuth* (in similar alloys) could be found by § 90, *b*, *c*; *antimony*, by § 96, *d*; *arsenic*, as in § 120, 6.

Litharge, Lead Dross, Hearth Bottoms.

123. *Oxides of lead, antimony, arsenic, bismuth, copper* (*zinc*) and

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iron, with *sulphates*, may all be present if the lead is made from mixed ores.

1. Sulphur is found by § 108, *c*.

2. Reduced alone on coal it yields a fusible metallic globule, from which *arsenic*, *antimony* and *zinc* reactions can be obtained; see raw lead, § 119. The button yields *copper* (sometimes also *nickel*,) by §§ 88, *f*, 2; and 78, *b*; this copper yields *silver* on cupellation with lead, § 119, 5.

3. A fragment treated in R. F. beside borax on coal yields a greenish to colorless glass and a metallic button. The glass oxidized on wire shows *iron* by its yellow color, at least while hot. (Any *cobalt* which might possibly be present in similar impure lead oxides would be also found in this borax, see German silver, § 121, 2.)

Paints.

124. 1. *White lead paint*, if pure, should contain only *carbonate* and *oxide of lead*, ground with oil, but it is often mixed with *oxide of zinc* (*zinc white*) and *barium sulphate* (*barytes*).

A little of the paint is gently heated on coal, to char the oil, after which the residue is treated in R. F. on coal, previously prepared with cobalt solution, so that any *zinc* is readily detected, § 79, *b*, *d*.

The residue of the paint is then treated with a strong R. F. so long as it yields any coat, after which the residue, consisting of barium sulphide and sulphate, if the latter was present in the paint, is tested on wire for *barium* by the flame test, § 42, being moistened with hydrochloric acid if necessary.

2. Colored paints may be examined for *copper*, *chromium*, *arsenic*, *iron*, etc., by the scheme, p. 150.

Colored paints, containing *lead* are readily tested for this metal as above. If they contain *zinc* and *barytes* these can also be detected as above, but the presence of *copper* would interfere with the flame test for the barytes. The *copper* is easily found by a reduction test with soda, after volatilizing the zinc and lead as before.

The quickest method to detect *barytes* in presence of copper would be to char the paint in a clay capsule, igniting the residue thoroughly, moistening it with a little dilute sulphuric acid, boiling the resulting mass in a test tube with strong hydrochloric acid and a drop or two of nitric acid, diluting, collecting the insoluble residue on a filter, washing it with hot water to remove all copper chloride, treating the residue on coal to expel lead as above, and testing the residue by the flame test for barium.

Iron can be readily detected in any paint, in absence of other metals whose oxides cannot be reduced from borax, as cobalt and chromium, by treating the residue from charring the paint, as directed in § 123, 3, adding a little lead. Even in presence of chromium any considerable amount of iron oxides could be detected by the magnetic properties which would be shown by the charred mass, if strongly reduced alone on coal.

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SCHEME OF BLOWPIPE ANALYSIS,

BY

PROF. T. EGLESTON.

THE substance may contain **As—Sb—S—Se—Fe—Mn—Cu—Co—Ni—Pb—Bi—Ag—Au—Hg—Zn—Cd—Sn—Cl—Br—I—CO^2—SiO^2—N^2O^5—H^2O—Te.** See Note, p. 162.

1. Treat on Ch. in the O. F. to find volatile substances such as **As—Sb—S—Se—Pb—Bi—Cd,** etc., § 33, *et seq.*; and also test in the open tube to find whether **As, Sb, S** are present as arsenides, etc., or in the oxidized state, § 32, *et seq.*

a. If there are volatile substances present, form a coating and test it with S. Ph. and tin on Ch. for **Sb,** § 96, *b*, or to distinguish between **Pb** and **Bi,** § 90, *a*.

b. If there are no volatile substances present, divide a part of the substance into three portions and proceed as in **A.**

α Yellow coat, yielding with S. Ph. a black bead; disappearing with blue flame, no part of it yielding greenish **Sb** flame, **Pb** and **Bi.**

β Yellow coat, generally with white border, yielding black or gray bead with S. Ph., disappearing with blue flame; also the border disappearing with greenish flame; **Pb** and **Sb.**

γ Yellow coat, very similar to *b*, but yielding no blue flame; **Bi** and **Sb.**

Make a special test for **Bi,** § 90, *b*. **Pb** in presence of **Bi,** if not in too small quantity, is to be detected by the blue flame yielded by the coat or by the reduced metal itself. See also § 86, *b*.

2. If **As—Sb—S—Se** are present, roast a large quantity thoroughly on Ch., § 43. Divide the substance into three portions, and proceed as in **A.** (See note on p. 162.)

A. TREATMENT OF THE FIRST PORTION.—Dissolve a very small quantity in borax on platinum wire in the O. F. and observe the color produced. Various colors will be formed by the combination of the oxides. Saturate the bead and shake it off into the porcelain dish; repeat this once or twice, § 44, 1, *c*.

a. Treat these beads on Ch. with a small piece of lead, silver, or gold, in a strong R. F., § 44, 1, *d*.

b. **Fe—Mn—Co,** etc., remain in the bead, § 44, 1, *d*.

If the bead spreads out on the Ch., it must be collected to a globule by continued blowing.

Make a borax bead on platinum wire and dissolve in it *some* of the fragments of the bead, reserving the rest for accidents.

c. **Ni—Cu—Ag—Au—Sn—Pb—Bi** are reduced and collected by the lead button, § 44, 1, *d*. (**Sn, Pb,** and **Bi,** if present, will partly volatilize.)

Remove the lead button from the bead while hot, or by breaking the latter, when cold, on the anvil between paper, carefully preserving all the fragments.

d. If **Co** is present, the bead will be blue.

If a large amount of **Fe** is present, add a little borax to prove the presence or absence of **Co,** § 74, *c*.

If **Mn** is present, the bead when treated on platinum wire in the O. F. will become dark violet or black.

e. If only **Fe** and **Mn** with no **Co** are present, the bead will be almost colorless.

Look here for **Cr, Ti, Mo, U, W, V, Ta** by the wet way. (A notable amount of **Ti** may be detected with S. Ph. and tin in the original oxides, in absence of other coloring non-reducible oxides, Tab. VI. **Mo** will be shown by the cloudy brown, or black appearance of the borax bead in R. F. on platinum wire, Tab. VI.)

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f. Treat the button *c* on Ch. in the O. F. until all the lead, etc., is driven off; **Ni, Cu, Ag, Au,** remaining behind; or separate the lead with boracic acid, § 78, *c*, 1.

g. Treat the residue *f* on Ch. in O. F. with S. Ph. bead, removing the button while the bead is hot.

h. If **Ni** and **Cu** are present, the bead will be green when cold, § 88, *f*, 1. If **Ni** only—yellow. If **Cu** only—blue.
Prove **Cu** by treating the S. Ph. bead with tin on Ch. in the R. F., § 88, *d*.

i. For **Ag** and **Au** make the special test No. 8.

B. Treatment of the Second Portion.—Drive off the volatile substances in the O. F. on Ch. Treat with the R. F., or mix with soda, and then treat with the R. F., for **Zn, Cd, Sn.** If a white coating is formed, test with cobalt solution; §§ 79, 94.

If **Zn** is found, it is not generally necessary to look for **Sn,** and vice versa, as they very rarely occur together. (See note, p. 162.)

C. Treatment of the Third Portion.—Dissolve some of the substance in S. Ph. on platinum wire in O. F., observing whether SiO^2 is present or not, and test for **Mn** with potassium nitrate, § 72, *b*.

3. Test for **As** with soda on Ch. in the R. F., or by special tests, § 98.

4. Dissolve in S. Ph. on platinum wire in the O. F. (if the substance is not metallic and does not contain any **S**), and test for **Sb** on Ch. with tin in the R. F., § 96, *b*. (To detect a little **Sb** with **Cu** or **Sn,** see § 96, *d*.)

5. Test for **Se,** § 102.

6. In absence of **Se** fuse with soda in the R. F. and test for **S** on silver foil, § 103, *c*. In presence of **Se** test for **S** in open tube, § 103, *b*. (To distinguish between **S** and SO^3, see § 103, *d*.

7. Test for **Hg** with *dry* soda in a closed tube, § 84.

8. Mix some of the substance with assay lead and borax glass and fuse on Ch. in the R. F., § 83, *b*. Cupel the lead button for **Ag.** Test with nitric acid for **Au.**

9. Test for **Cl, Br** and **I** with oxide of copper, §§ 104, 105, 106.

10. Test for **Br** with bisulphate of potassa, § 105, *d*.

11. Test for H^2O in a closed tube, § 30, *d*.

12. Test on platinum wire, or in platinum-pointed forceps, for coloration of the flame, § 42.

13. Test for CO^2 with hydrochloric or nitric acid, § 111, *a*.

14. Test for N^2O^5, § 110.

15. Test for **Te,** § 101, *c*.

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Note.—The scheme is to be varied slightly according to circumstances. If sulphides, arsenides, etc., are under treatment, the substance must be carefully roasted; but if test 1 fails to show S, As, Sb, or Se, as sulphides, arsenides, etc., the substance is either an oxide or an alloy. If an oxide, the roasting, 2, is omitted. If an alloy, it is subjected to the test 1, *a*, for Pb, etc., and then the test 2, A, is performed by fusing the alloy on coal with borax in the R. F., thus combining 2, A, and 2, A, *a*, in one operation. Some sulphides during the roasting 2, A, will reduce to metal, and then after thorough roasting are to be treated like alloys. A metal or raw sulphide, etc., must never be treated on platinum wire, but the metal is fused on coal with the flux ; in R. F., if it is desired to get only non-reducible metals, as Fe, Co, etc., in the flux ; in O. F., if Cu, Ni, and other reducible metals are to be fluxed. The flux is then transferred to the wire. Sulphides, etc., must always be roasted before testing with borax and S. Ph.

The word bead always refers to the flux, and button to the metal. In regard to 2, B, Sn can always be found in the presence of Zn by reducing the oxides with soda and a little borax, and triturating the mass with water, p. 69. In certain alloys, *e. g.*, bronzes, containing both Sn and Zn, the Zn can be detected by treating a short time in R. F. and testing the coat formed with cobalt solution, as the Zn is more volatile and comes off first.

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CHAPTER V.

ANALYSIS WITH THE AID OF THE WET WAY.

125. It is not intended here to give any complete directions for wet analysis, but only for the detection of the substances most commonly met with in examining ordinary ores and metallurgical products, under conditions which do not admit of their detection B.B. alone. The methods are intended to be general and will naturally often be capable of modification by the more experienced analyst.

126. *Lead.* Bismuth interferes sometimes with its detection. Make a reduction test with soda, dissolve the resulting metallic residue in nitric acid, evaporate to dryness, warm with dilute sulphuric acid, allow to stand until quite cold, filter out the insoluble lead sulphate, wash it with a little dilute sulphuric acid and test it for lead. The bismuth will have passed into the acid solution, and can be precipitated by excess of ammonia. If the substance is soluble in nitric acid the reduction may be omitted.

127. *Cadmium.* Arsenic may interfere with the detection of cadmium even in the closed tube. Fuse the finely divided substance in a porcelain crucible with five or six times its volume of potassium nitrate, boil the mass with water, filter and test any insoluble residue for cadmium on coal and in the closed tube.

128. *Antimony.* It may not be detected with certainty in the presence of both bismuth and tellurium. Boil the finely divided substance with hydrochloric and nitric acid together, evaporate to expel excess of nitric acid, dilute slightly with water, filter, if necessary, and add more water, but if any considerable turbidity now ensues add hydrochloric acid to redissolve the precipitate, saturate with hydrosulphuric acid, digest the precipitate with warm, yellow ammonium sulphide, filter, dilute with water, acidify with dilute hydrochloric acid, collect any precipitate, wash it thoroughly with water containing a little hydrosulphuric acid, dry it, fuse it in a porcelain crucible with sodium carbonate and nitrate, dissolve out the soluble matter with a little cold water, wash the residue on a filter with a little dilute alcohol and test as in § 96, *a*.

129. *Zinc.* Its detection may be rendered uncertain by the pres-

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ence of tin. Dissolve the finely powdered substance in hydrochloric and nitric acids, expel excess of nitric acid, dilute with water, but leave the solution decidedly acid, saturate at once with hydrosulphuric acid, without first filtering, filter out any precipitate which now forms, boil the filtrate to expel hydrosulphuric acid and then boil again with excess of sodium carbonate. Any precipitate which forms is filtered, washed, dried and tested for a zinc coat with soda (and borax if necessary).

130. *Chlorides, bromides, iodides and cyanides.*

To the solution to be tested add silver nitrate, filter out and wash any precipitate insoluble in dilute nitric acid, and test a portion of it for cyanogen by § 108, *b*. (Before adding the silver nitrate, any sulphides present should be decomposed by adding copper sulphate solution, and the resulting copper sulphide filtered out.)

If cyanogen is found, ignite the rest of the silver precipitate in a porcelain crucible and afterwards fuse it, or the original precipitate free from cyanogen, with sodium carbonate, boil with a little water, filter and divide the solution. To one part add a few drops of dilute sulphuric acid and then a little starch paste (made by boiling a very little starch with water). Add a drop or two of fuming nitric acid, which will cause a blue or very dark purple color, according to the amount of iodine present. Next add chlorine water * drop by drop, until the iodine reaction disappears, and then a drop or two more (an unnecessary excess must be avoided) to liberate any bromine present. If there is not enough of the bromine to produce the brownish-red or even pale reddish yellow tint, characteristic of free bromine, add two or three drops of chloroform or carbon bisulphide (ether is not so good), shake well, and set aside until the chloroform, now colored by the free bromine, collects at the bottom.

To the other part add silver nitrate in slight excess, filter, shake the precipitate with a little ammonia diluted with three volumes of water, filter out any undissolved residue of silver iodide, acidify the filtrate with nitric acid, collect, wash and dry the precipitate, fuse it with sodium carbonate, dissolve in water, filter, nearly neutralize with sulphuric acid, evaporate to dryness, fuse with potassium bichromate, pulverize the mass and heat it gently with a little strong sulphuric acid. Chlorine will be indicated by brownish-red fumes, which are distinguished from any liberated bromine by the yellow color they impart to a drop of ammonia cautiously introduced into the midst of the fumes on the end of a glass rod. The colored fumes consist of chromic oxychloride.

131. *Iron, aluminium, barium, calcium, strontium, magnesium and potassium.*

a. 1. *Substances soluble in water or in acids.*

* Made by heating binoxide of manganese with a little hydrochloric acid in a test-tube, provided with a cork through which passes a bent tube. One end of the tube stops just below the cork in the test-tube, the other end dips into a little water in any vessel, so that the water may absorb the chlorine gas evolved from the acid and binoxide.

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After separating from the solution, which must be quite acid, the metals that can be thrown down by hydrosulphuric acid, the solution is boiled to expel the remaining hydrosulphuric acid, a few drops of nitric acid are added, and the solution again boiled, after which ammonium chloride and excess of ammonia are added, the solution is boiled, filtered if a precipitate forms, and the filtrate (or solution free from any precipitate) kept for *b*, while the precipitate is treated as follows: It is washed, dried and fused with three volumes of sodium carbonate and three of potassium nitrate, the mass boiled with water, the solution filtered off from any residue, acidified with hydrochloric acid, ammonia added in slight excess, and any precipitate which forms, even after some time, filtered out, dried and tested for alumina with cobalt solution. (If silica was present originally, in a form soluble in acids, the above solution should be evaporated to dryness, the residue heated with a little hydrochloric acid, diluted and filtered off from the insoluble residue, before adding the ammonia.)

The residue insoluble in boiling water is tested for iron with borax.

2. *Substances insoluble in acids.*

Plattner's general directions for their decomposition are as follows: Mix seventy-five to one hundred milligrammes of the finely divided substance with its weight of powdered borax glass and as much soda (two parts of borax if much magnesia or alumina are present, and more soda also in presence of much barium sulphate) in an agate mortar, adding a silver or gold button of sixty to eighty milligrammes, if small quantities only of reducible metals are present; the whole is wrapped in a soda-paper cylinder, § 147, *a*, and reduced just as directed for the copper assay, § 166. When reducible metals are known to be absent no gold or silver button need be used. The fusion must be made with care and continued until the melting glass is fluid and free from bubbles or reduced metal. When cold the glass is broken between paper, the metallic button, if present, removed and the finely powdered glass well moistened with water and heated with dilute hydrochloric acid. After evaporating it to dryness over a water bath, and heating it there until it scarcely gives any acid odor, it is heated with a little hydrochloric acid and a few drops of nitric acid, and finally with water. The insoluble silica, with perhaps some barium sulphate, is filtered out and the bases are detected as in *a* above.

b. The ammoniacal filtrate is treated with ammonium carbonate unless a drop or two of it gives a precipitate with ammonium sulphide, in which case the whole solution is boiled and the sulphide added so long as it forms a precipitate, the precipitate is filtered out, and the solution now treated with ammonium carbonate, unless it is still colored brown by some dissolved nickel sulphide, in which case it is made slightly dilute with hydrochloric acid, the black nickel sulphide filtered out, the solution made ammoniacal again, and finally treated with ammonium carbonate. Any precipitate now obtained is filtered out and the filtrate reserved for *c*.

The precipitate is washed, dissolved in the least possible quantity of acetic acid, slightly diluted, boiled and treated with potassium bichromate so long as this causes any yellow precipitate, which indicates barium, the precipitate being barium chromate. The filtrate from this precipitate, or the solution free from any precipitate, is treated again with ammonium carbonate in excess and boiled. If a precipitate forms it is collected, washed, dissolved in a very little acetic acid, and treated with potassium sulphate solution (one of the potassium sulphate to

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two hundred of water). After a few hours any strontium will separate as sulphate, and the filtered solution is made alkaline with ammonia and treated with ammonium oxalate, which will throw down any calcium as a white oxalate.

c. The filtrate is divided and one part is tested for magnesium by adding solution of S. Ph., which will precipitate magnesium ammonium phosphate. The other part is evaporated to dryness, ignited to expel salts of ammonium, the residue dissolved in a little water and tested for potassium by the flame test.

Note 1. The above process is not designed to be used in presence of phosphates, borates, or fluorides. These can in general be identified by determinative mineralogy and the processes they require are too extended for this work.

Note 2. Barium and strontium sulphates may remain in part with the silica, as may some calcium sulphate. Fuse the silica residue with six volumes of soda in a platinum crucible, extract all soluble matter with boiling water and test the insoluble residue as in *b*, beginning with the first use of acetic acid.

132. *Special examination of barium, strontium and calcium sulphates.*

Calcium is easily detected in all cases, because it remains on the coal as an infusible coating, when the finely powdered mineral is fused with soda for some time in a strong O. F.; the barium and strontium oxides finally sink into the coal with the soda. The calcium can be further tested by dissolving it in hydrochloric acid, diluting slightly, adding a drop or two of dilute sulphuric acid, filtering out any barium or strontium sulphates, after some time, and adding ammonia and ammonium oxalate, to throw down the calcium as oxalate.

To detect a little *strontium* in *barium carbonate*, dissolve this in hydrochloric acid, evaporate to dryness, add a drop or two of water, and then a moderate quantity of alcohol. The strontium red flame will be seen on setting fire to the alcohol and stirring it; or more plainly by dipping a little cotton wrapped on platinum wire into the mixture and setting fire to it.

Barium sulphate is tested for *strontium* by fusing a paste of it in fine powder with a little powdered graphite and water on coal in R. F., adding hydrochloric acid to the mass in a dish and then proceeding as above.

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CHAPTER VI.

SPECTRUM ANALYSIS.

133. The frontispiece shows the spectra of various elements in the detection of which the spectroscope lends great aid. A simple direct vision pocket spectroscope can be made very useful, if the student first acquaints himself with the spectra of the various metals, using generally their chlorides for the purpose. The salt is brought into the non-luminous flame of a Bunsen burner (an alcohol flame is less useful) on a thin platinum wire and the resulting colored flame directly observed with the spectroscope.

If the spectroscope carries no scale, and the student has not salts of all of the above metals at hand for comparison, much may still be done by simultaneously introducing into the flame another wire carrying some known salt whose lines will serve as reference lines to determine the approximate position of those given by the substance under examination. Salts of potassium, lithium, and strontium will be found convenient for this purpose, while the sodium line is distinct in almost every spectrum. Naturally, when any of the three metals named is itself sought for its salt cannot be simultaneously used for reference. If the pocket spectroscope has a little comparison prism, then any reference salt can be simultaneously used, since the two substances can be heated in two different flames, producing two spectra, one above the other, the corresponding lines of any metal present in both spectra falling in the same vertical line. If the spectroscope has a scale, which is sometimes the case with the pocket spectroscopes and always should be with the larger ones for laboratory use, then the position on this scale of the lines of any spectrum can be fixed once for all, provided some definite line, as Na α, is always brought to the same point on the scale of the instrument. It is well thus to locate the principal lines of each spectrum on a paper scale corresponding in its divisions to the scale of the instrument, but this is not absolutely necessary, since by determining certain lines, as K α, Li α, Na α, Sr δ, and K β, and calculating for each interval between these separately, the positions of the principal lines of the other metals, the student can lay down on his paper scale

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the latter lines very nearly in their correct positions. The line K α may come at the right or left hand of the field of view, according to the construction of the spectroscope, and the numbers in the scale above the potassium spectrum in the frontispiece are no strict guide to the numbers on the scale of any given instrument.*

134. Any soluble salt, and often insoluble compounds, may be examined spectroscopically. If the insoluble substance, especially after ignition, is moistened with a drop of hydrochloric acid the flame coloration is often intensified.

Barium and *strontium* sulphates will yield a good spectrum if first ignited in R. F. and then moistened with the acid.

Alkalies can be found in insoluble silicates by two methods. They may be decomposed by J. L. Smith's method, viz.: heating in fine powder to bright redness for half an hour or so in a platinum crucible, with six or eight parts by weight of calcium carbonate† and about one of powdered ammonium chloride (sublimed first, if it leaves any residue when ignited on platinum foil) digesting in hot water, filtering, treating the filtrate with ammonia and ammonium carbonate, filtering, evaporating the filtrate to dryness, igniting and dissolving the residue in a very little water, which is then tested on wire before the spectroscope.

The second method consists in igniting them in a platinum crucible or on foil, to low redness, with a large excess of ammonium fluoride and simply testing the residue, brought upon the wire with a little water.

Barium, *strontium* and *calcium* can be obtained in soluble form from silicates or sulphates by fusing them with a large excess of soda, extracting the soluble salts from the fused mass with boiling water, washing the insoluble residue and testing it at once, after dissolving it in hydrochloric acid; if very much iron or alumina are present they may be separated by adding ammonia, filtering, and precipitating the alkaline earths as carbonates with ammonium carbonate. The carbonates are then moistened on wire with hydrochloric acid and examined with the spectroscope. In presence of very much lime

* Steeg and Reuter, Homburg vor der Höhe, Germany, make an excellent single prism spectroscope, with observing telescope, collimating lens with slit and independent scale tube, with accessory apparatus, for 100 marks.

† Made by dissolving calcite in dilute hydrochloric acid, keeping the calcite in excess, warming, stirring in powdered and very strongly ignited calcite, until the solution reacts alkaline, filtering, heating nearly to boiling, adding ammonium carbonate solution in excess, filtering, washing the precipitate thoroughly and then drying it. Finely powdered calcite may be used at once, if found free from the alkali metals.

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it is well to dissolve the mixed carbonates in nitric acid, evaporate gently to dryness, dissolve out the calcium nitrate with alcohol and test the dried residue at once for barium and strontium. If there is too much barium to admit of the detection of traces of strontium, ignite the residual nitrates with much ammonium chloride and extract the resulting strontium chloride with alcohol, leaving barium chloride behind.

Thallium is detected generally with ease in pyrites, flue dust, etc., containing it, by simply heating the raw substance in the flame. Fresenius recommends separating the bulk of any crude sulphur, which is to be tested for thallium, by dissolving it in carbon bisulphide. In presence of very much sodium the thallium line may only flash out at the first moment of heating the moistened substance.

Indium. The mineral to be tested is dissolved in a suitable acid, nitric and hydrochloric combined are best for sphalerite; the solution, filtered from any insoluble residue, is heated with ammonia in excess, the precipitate filtered out, carefully washed, and tested at once on wire, after being mixed with a little hydrochloric acid. At least the line In α will be seen, if any noteworthy amount of indium is present.

Various chlorides and a number of other bodies yield more or less distinct spectra, but not generally sufficiently well defined to be of use for their detection. *Cupric chloride* yields, however, a very well marked spectrum containing many lines in nearly all parts of the field of view; *manganese chloride* also gives some fairly conspicuous lines.

Absorption spectra, obtained by holding transparent media before the spectroscope slit and allowing the light from a luminous flame, or the daylight, to pass through them into the spectroscope, are occasionally of interest to the chemist. The continuous spectrum yielded by ordinary luminous flames will be interrupted by dark lines or bands, whose position is characteristic of different bodies. Thus didymium salts, in solutions so weak as to show no color, present a distinct absorption spectrum.

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CHAPTER VII.

QUANTITATIVE BLOWPIPE ANALYSIS (ASSAYING).

I. GENERAL REMARKS.

A. Apparatus.

The following apparatus for quantitative work is needed, in addition to much of that described for qualitative work.*

135. *A balance.* One indicating with certainty $\frac{1}{10}$ milligramme will suffice for all assays except for gold and silver, and will answer also for weighing out charges for these metals, the weight of the buttons obtained being afterwards determined on a more delicate balance, or by measurement. A balance indicating only $\frac{1}{2}$ milligramme will even suffice for approximate assays of the less precious metals or for weighing out charges for the precious metals.

When silver buttons are to be weighed a very delicate balance is necessary, one whose index needle deviates at least one division of its scale for $\frac{1}{10}$ milligramme. By averaging the readings of several vibrations of the beam, using a lens for the reading, a close approximation to $\frac{1}{25}$ milligr. may be made. Measurement, however, gives better results with quite small buttons than any weighing, while gold buttons from blowpipe assays cannot ordinarily be weighed.

A. Lingke & Co., Freiberg, Saxony, make a balance expressly for quantitative blowpipe work. The scale pans are hung by silk threads and the balance is provided with a folding glass case. The general arrangement of the balance is shown in Fig. 26. As now constructed the best balance is set up on a solid block and not on its packing box, as represented in the figure, although cheaper ones are still so made. The beam and its support are so hung that they are kept in the proper level by the swinging tongue *b*, when the beam is raised by turning the knob on the front of the block below, which winds

* The apparatus and reagents marked † in §§ 10 *et seq.* are needed for quantitative as well as qualitative work; especial quantitative reagents are described in § 148. The student can best determine what reagents may be needed on a trip by reading the descriptions of the several assays.

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up the cord *d*, running over three pulleys suitably placed. The camel's hair brush *f* prevents undue swinging of the tongue *b* when the beam is raised. The beam is 180 millim. long and all the metal parts are gilded, while the knife-edges rest on agate bearings. The balance costs, with case, weights, apparatus for determining specific gravity, brush, ivory spoon, etc., 144 marks, about $36, in Freiberg. It will weigh accurately and rapidly to $\frac{1}{10}$ milligr. when carrying loads of one gramme. The index needle of one in the author's laboratory deviates one division on the scale for $\frac{1}{10}$ milligr. when carrying such small weights as the silver buttons from blowpipe assays, and can therefore be used for ascertaining the weight of such buttons to $\frac{1}{20}$ of a milligr.

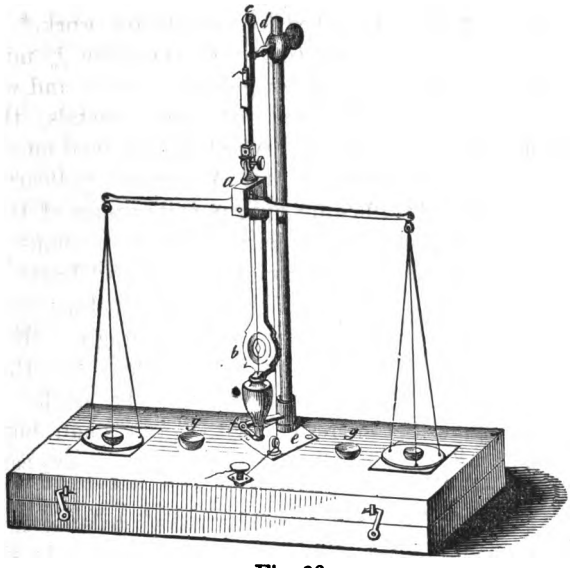

Fig. 26.

Becker & Sons, New York, also make a blowpipe balance, of excellent finish, enclosed in a glass case, the index needle deviating one division for $\frac{1}{20}$ milligr.

It is scarcely necessary to remind the operator that such balances must be protected from moisture by placing fused calcium chloride or ignited potassium carbonate, in a suitable vessel, within the case, and must be protected from dust and occasionally cleansed. They also require very careful handling. All blowpipe balances should be provided with horn capsules for weighing out fluxes, etc., while the highly polished or gilt metal capsules are to be used only for weighing ores and buttons. Nothing but clean metal buttons,

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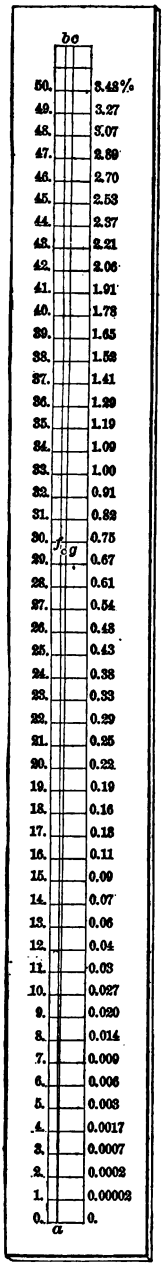

Fig. 27.

or the like, should ever be weighed directly on the scale pans of any good balance.*

136. *Weights.* The weights should run from 1 gramme down to 0.1 milligramme. The gramme (grm.) is very nearly equal in weight to 15.432 grains, and is equal exactly to 1000 milligr., 100 centigr. or 10 decigr. The decigramme (or 0.1 grm.) is the blowpipe centner (ctr.). When a charge of 1 ctr. of ore, etc., is taken each milligr. of metal obtained from it represents one per cent. It may be well for the student to remember that there are 29,166.66 troy ounces in a ton of 2000 pounds avoirdupois, so that each milligramme of silver or gold, for instance, obtained from a centner of ore, represents 291.66 ozs. troy per ton of ore.

137. *Plattner's Scale.* This consists of a flat ivory plate, Fig. 27, on which are two convergent lines, *a b* and *a c*, separated at a distance of 156 millimeters from *a* by a space of one millim. These lines must be exceedingly fine and true, or the scale will be worthless. The author has rarely seen any reliable ones made in this country, because the lines were too coarse. Lingke's are always well made and his later ones show, as in Fig. 27, on the right hand side of the scale, the weight of a silver button in milligrammes, or the percentage of silver, if one centner of ore was used to obtain the button. The transverse lines, numbered on the left hand side of the scale, from 0 to 50, are to be used with the Table, p. 207, from which the weight of a gold button may be read in milligrammes. Should a button be found to come between two transverse lines, as at *f g*, its weight would be found by subtracting the next lower figures, on the ivory scale for silver, or in the Table, p. 207, for gold, from the next higher ones and adding the proper amount of their difference to the lower figures. In this case, for silver, there would be indi-

* A balance, similar in design to the balance, Fig. 26, but less delicate, is sold at a very reasonable rate by Eimer and Amend, New York. It serves very well for prospecting work and for the assays of base metals.

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cated 0.67 + 0.04 (one-half of the difference between 0.67 and 0.75) = 0.71 per cent. of silver.

Plattner's scale formerly had figures on the right hand side running down from 122.5 at the transverse line 50 to 0.0009 at the line 1. Such a scale, originally designed for use with an old system of weights, can still be used for the gramme weights by means of the left hand numbers of the transverse lines and the right hand figures of the scale shown in Fig. 27, for silver, or the Table, p. 207, for gold, since these lines are the same both in the old and new style of the scale made by Lingke.

For further directions in regard to the use of this scale and for other means of measuring buttons, see § 154.

138. *Artificial Coals,* as *square coals,* coal crucibles* and *coal capsules.*

a. The manufacture of these, while it may at times be necessary, requires considerable experience and the articles can always be purchased ready made. In making them, take one part of starch powder and six of water, mix the starch to a thin paste with a part of the water, and stir the rest of the water boiling hot into this, until the starch is all converted into a gummy paste. To this add by degrees charcoal dust, rubbing the whole in a mortar, until the paste is too stiff to take up any more dust, and then knead and work in more coal dust with the hands, until the whole is stiff and plastic. Articles moulded from this mass are gradually and thoroughly dried and then heated in a roomy covered crucible to low redness, small articles being thus heated in a porcelain crucible over the spirit lamp. When combustible gases cease to escape from the crucible and the upper row of articles in the crucible glow slightly, the heating is ended.

b. Coal crucibles are made in the mould, Fig. 35, using a wooden stamp, Fig. 28, the diameter of which is 27 millim. at *a b*, and 9 millim. at *c*. The mould is filled with a ball of coal paste, rolled in coal dust, and the stamp, also dipped in coal dust, is pressed upon it and drawn out by carefully turning it around. The mould is then taken apart as described in § 139, and the crucible with a cavity 6 millim. deep, Fig. 29, is dried and charred.

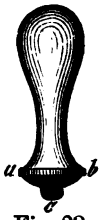

Fig 28.

c. Coal capsules, Fig. 30, are made in a similar way in

* Square blocks of sound, soft charcoal may be used, when necessary, in place of these artificial coals. The holes for crucibles, etc., are to be bored in a cross section of the coal.

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Fig. 29.

the mould, Fig. 33, using a special stamp of boxwood, Fig. 31, whose moulding part is a segment of a sphere, having a radius of 20 millim. A strip of paper is first put into the mould, by means of which the finished capsule can be lifted out.

Fig. 30.

d. Square Coals and Covers. (Fletcher's clay furnace, § 147, *g*, 3, is a good substitute for these when gas can be used.) The coals are made in a mould of hard wood, consisting of four pieces as shown in Fig. 32. The mould being put together and the smaller block of wood *D* put in it and covered with a square of paper, the mould is powdered inside with coal dust and filled with coal paste, into which the wooden stamp *A*, after being dipped in coal dust, is pressed, carefully turned around and drawn out. The ring *g* is unclamped, and the square coal *F* removed, dried and charred.

Fig. 31.

Covers are made in the same mould after placing the block *E* upon *D*, using the wooden stamp *B*. *G* shows a cover. The prismatic space in the mould is 40 millim. deep and 35 millim. square. The block *D* is 9 millim. high, the block *E* is 17 millim. high, and both fit the square hole exactly. The rim *c d* of the stamp *B* fits exactly within the guide pins on *C* and the projection *e* of the stamp *A*, at its base, has a diameter of 22 millim. and is 18 millim. long. The lower spherical part *f* of the stamp *B* is 9 millim. thick and is 22 millim. wide where it joins the flat part *c d*.

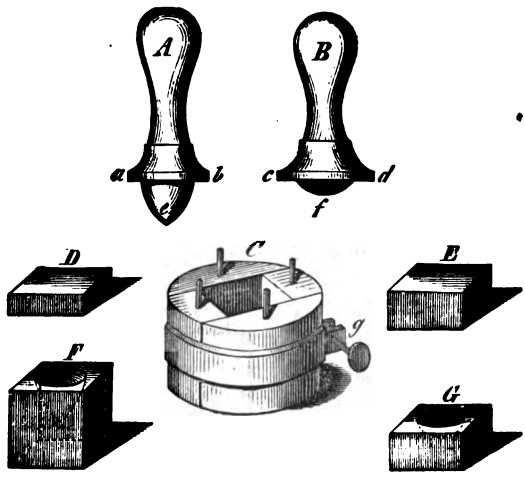

Fig. 32.

139. *Clay Crucibles and Capsules.* These can be purchased, but they are sometimes made so thick as to be useless. Much skill is needed in their manufacture. Good fire-clay is carefully elutriated and the very fine dust thus obtained is worked with a little water into a tough, plastic mass, the proper consistency of which can be

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learned only by experience. It must be worked until it will retain its shape after being removed from the mould, when the crucibles and capsules are not more than 0.8 millim. thick.

Clay Capsules are made in a box-wood mould well soaked with oil, Fig. 33, the spherical cavity of which is 20.5 millim. wide and 7 millim. deep, while the spherical projection on *B* has a radius 0.8 millim. shorter. The mould and stamp are slightly oiled, a strip of paper 50 millim. long and 5 wide is put into *A*, a ball of clay about 12 millim. in diameter is also pressed into it with the fingers and then carefully moulded with *B*, which is withdrawn by turning it so as not to distort the shape of the capsule, after which the superfluous clay is cut away. The capsules, Fig. 34, must be uniform in thickness and not over 0.8 millim. thick, before being baked. They are dried in warm air and finally baked at a red heat in an uncovered clay or platinum crucible, becoming still thinner in baking.

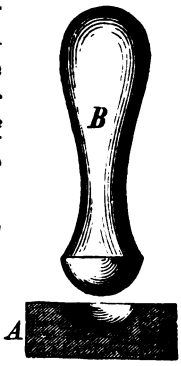

Fig. 33.

Fig. 34.

Clay Crucibles are made in the brass mould, Fig. 35, consisting of two halves which are held in place by the ring *C*. The stamp *A* carries at its lower end a blunt cone, 19 millim. in diameter above and 14 millim. long, with four conical openings, *a a*, through which the superfluous clay can pass. The mould itself is 0.8 millim. distant from this cone, all around, and the corners are filed away where its two halves join, as at *b*, so that some clay may catch in them and hold the crucible fast while the stamp is being turned and withdrawn. A ball of clay having been left to dry until it can with difficulty be pressed together between the fingers, is placed in the mould, previously oiled and set within the ring on the anvil, which rests upon several folds of woolen cloth. The oiled stamp is then driven into the mould with a mallet until its rim *c* meets the edge *d* of the mould. To remove the crucible from the mould, this is pushed upward out of the ring *C*, one of the halves held in each hand, and one half pushed down a little, until loosened from the crucible and then slightly pressed against it, while the other is similarly loosened. After cutting off the outer projections made by the cavities at *b*, the crucible is air-

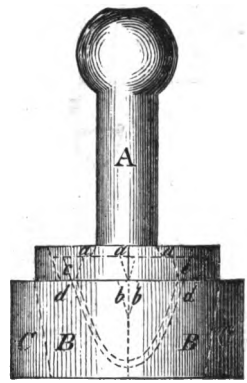

Fig. 35.

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dried and then baked. Fig. 55, § 148, *j*, shows a clay crucible (but there lined with charcoal).

140. *Coal Crucible Holder.* This is a cylinder about 60 millim. long and 25 thick, Fig. 36, with different shaped cavities in the ends to hold coal crucibles and capsules. It must be porous and may be made of pumice stone, or of clay and coarse coal dust, the latter burning away when the clay is baked in a partly covered vessel.

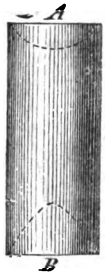

Fig. 36.

141. *Clay Crucible Stand.* This consists merely of a metal ring or band of proper height, which may simply be cut from a piece of gas pipe of $\frac{5}{8}$ inch bore and $\frac{3}{4}$ inch high, on which the hot crucible or capsule can be placed when necessary.

142. *Square Coal Holder.** This is a square sheet-iron box, open above and below, Fig. 37, in which the square coals are held by turning the screw *c* by the handle *e*, thus advancing the clamping plate *d*. See also Fig. 62. Each face is 32 millim. wide and 36 high, and the front *B*, opposite the handle, has in the middle a hole, *a*, 7 millim. in diameter, and a slit *b*, running to the top, which can be closed by the door *h*. On one side is a slit *i*, 8 millim. long and 0.8 wide, immediately under which is a socket *k*, to hold the platinum wire mentioned below. The clay crucible or capsule is supported by a platinum wire† 0.6 or 0.7 millim. thick, and about 83 millim. long, bent as shown in Fig. 38. First the wire is bent to a ring *A* at one end, then the dotted end *l* is bent out at a right angle to its first position and finally shaped as shown by *B* below. The length of the upper horizontal part must be carefully adjusted, so that the ring may come exactly in

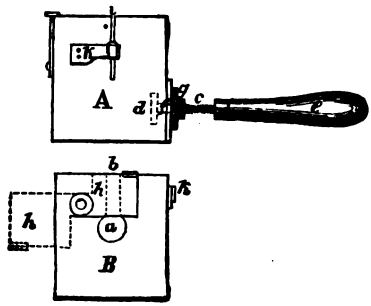

Fig. 37.

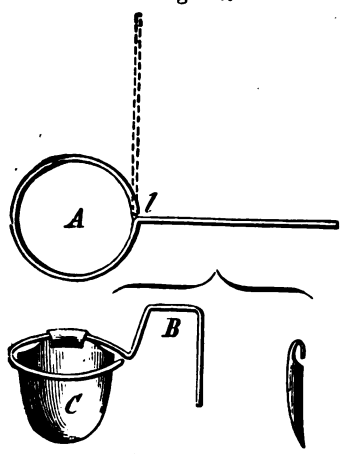

Fig. 38.

* The holder and coal fitted in it constitute a little charcoal furnace.

† The platinum wire is generally used only for supporting clay capsules, being

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the middle of the cavity in the square coal, as shown in Fig. 39. This coal (artificial or, as shown in the figure, ordinary charcoal) is passed upward into the holder so that the upper side, on which the part *B* of the wire rests, reaches exactly to the bottom of the slit *i*. A *very thin* platinum shield *C*, is hung on the wire opposite the opening *a*, to protect the coal, but only when roasting ores in capsules.

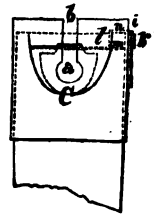

Fig. 39.

143. *Coal Borers;* one *square borer*, having two crossed cutting edges, each 8 millim. long, Fig. 40; the other *conical*, with four cutting edges, one continuous, the others interrupted, as in Fig. 41; it measures 22 millim. in diameter above, and is 18 millim long. When used on the square coals, § 138, *d*, this borer is sunk into the coal, until the upper side *a* is exactly level with the upper side of the coal.

Fig. 40.

144. *a. A Steel Spatula*, very highly polished, 95 millim. long and shaped as in Fig. 42. It must be kept scrupulously clean and bright.

Fig. 41.

b. An Agate Mortar, the bowl being about 50 millim. inside diameter. It is easily cleaned by rubbing with bone-ash, but must not be scratched with coarse fragments of very hard substances, which should first be crushed quite fine on the anvil or in the steel mortar.

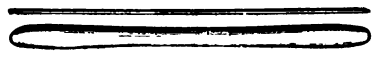
Fig. 42.

c. Steel Button Pliers, Fig. 43, with flat faced, delicate, close-shutting, slightly roughened jaws, to remove gold and silver buttons from cupels.

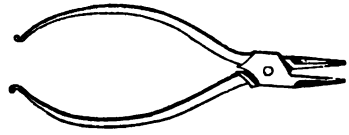
Fig. 43.

d. Forceps, of brass or German silver, Fig. 44, with close-shutting, fine points, which will easily and securely seize small buttons; they are used only for this purpose and to handle small weights.

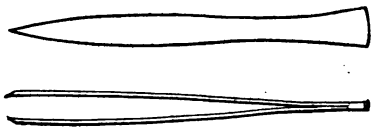
Fig. 44.

replaced by slightly stouter iron wire when crucible assays are made, in which there is danger of the charge overflowing and injuring the platinum wire.

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145. *Cupel Mould, Stamp and Stand.* The moulds are of iron,

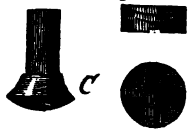

Fig. 45.

Fig. 45, *A*, 17 millim. in diameter, concave above, leaving a rim stout enough to bear moderate blows, and with two crossed slits in the bottom to admit the tips of the brass forceps, Fig. 12, § 10, *b*, with which the moulds are to be handled. The concavities are roughened inside to hold the bone-ash better. Two such moulds are desirable. The stamp *C*, of highly polished, hard steel, has at its lower end a convex projection, the segment of a sphere, somewhat larger in diameter than the corresponding concavity in the mould. The wire *a* of the cupel stand, Fig. 46, should pass through a metal collar, and be free from the wooden handle inside except at the bottom, having plaster cement between the wire and the collar.

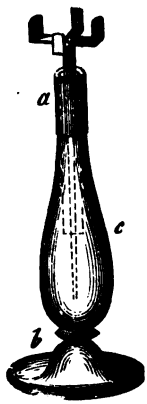

Fig. 46.

To make a cupel, press the mould full of bone-ash, set it on the anvil, place the stamp upon the bone-ash and strike it a few firm blows, not too heavy, with the hammer.

146. *a. Mixing Capsules,* Fig. 47; one of brass, gilt inside, or of German silver, highly polished; the other of horn. Both are shaped alike, 62 millim. long, the widest part of the bowl 24 millim., the deepest part 8 millim.; the lip 8 millim. wide, where it begins to round off, and 5 deep. The bottom should not be rounded up toward the lip end, but should be nearly straight, as in the figure, and the whole must be arranged so that powders can be easily and safely poured from it into a soda paper cylinder, § 147, *a*.

Fig. 47.

b. An *Ivory Spoon,* Fig. 48, finely polished; the bowl about 10 millim. long, 6 wide, and 2 deep, with not too thick a rim, especially at the point. The size of the bowl is important, since the ivory spoon serves as a rough measure for fluxes, and its shape is important, because a thick-edged, clumsy spoon is inconvenient to use.

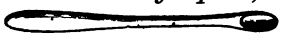
Fig. 48.

c. The *Brass Forceps,* Fig. 12, § 10, *b*, are needed in quantitative work.

d. A *Sieve,* Fig. 49, generally made of thin brass, for sifting test-lead. Its bottom is pierced with holes about 0.7 millim. in diameter, large enough to admit a pin of medium size. The sieve may be of a size to receive the mould, Fig. 33, when packing.

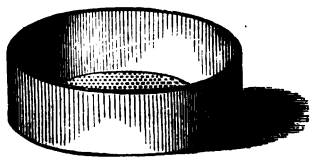
Fig. 49.

e. A *Test-lead Measure,* consisting of

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a glass tube, Fig. 50, with a sliding wooden plug, tipped with cork inside, and graduated to deliver 5, 10, 15 and 20 centners of test-lead, sifted through the above sieve. The tube should be about 45 millim. long and 5 or 6 millim. inside diameter. If the lead is obtained in fine powder, as in § 12, 9, the measure must be graduated also for this lead, since sifted, granulated lead will not occupy exactly the same volume for the same weight.

Fig. 50.

147. *a. Soda-paper Mould.* A wooden cylinder, 25 millim. long and 7 millim. thick, Fig. 51, with a handle. The soda-paper, *A*, § 148, *i*, is held as shown, the shorter edge parallel with the cylinder *B*, and is then tightly rolled around the latter, after which the projecting end of the paper is pressed down successively in several places over the end of the cylinder and firmly closed by pressing it against the table. To introduce a charge into this paper cylinder from the mixing capsule, the cylinder is held between the first two fingers of the left hand, its lower end resting on the third finger. The lip of the mixing capsule is then passed into the cylinder and held there by the thumb and fore-finger of the left hand, while the capsule is gently tapped with the brass forceps or ivory spoon, held in the free right hand. The charge, if containing quite loose materials, as dry ore, borax and test-lead, in the silver assay, will slip readily down into the cylinder, the adhering dust only being brushed in with the camel's hair brush, which must never be dipped into the whole charge. Charges containing soda, Plattner's flux, etc., must be pushed down with the handle of the ivory spoon, since they do not slide freely, and then the capsule is brushed clean.

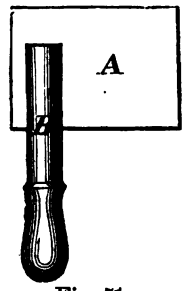

Fig. 51.

The capsule is now removed, the cylinder still resting on the third finger of the left hand, and the empty top of the cylinder pressed together with the forceps, this flattened end folded down upon itself twice or more with the forceps and thus rolled up, until the full part is reached, after which the folds are pressed down upon the top of the cylinder, a crease made across their top with the finger nail and the corners or ends pinched a little upward and together, so as to make the whole tight and compact.

b. A stiff *Button Brush*, to cleanse silver and gold buttons, after cupellation.

c. A small *camel's hair brush*, to clean scale-pans, mixing capsules, etc. The brush itself should be cut off nearly square, not pointed,

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and the hairs may project about 13 millim. from the quill holder, which should be about 3 millim., or a little more, in diameter at the brush end. Larger brushes may be used for cleaning the balance, but are not good for brushing residues of charges, etc.

d. A *touch-stone* will be sometimes useful in testing the precious metals or their alloys. It consists of hard black basalt, jasper or silicious slate and is cleaned by oiling its surface, rubbing it with charcoal and wiping it off.

e. A *platinum crucible*, very light, § 189, *b*, will often be useful.

f. An *ore sieve*, of brass wire gauze, 80 meshes to the linear inch, for sifting ores.

g. Automatic Apparatus. 1. *Blowpipe stand and hand-bellows.* For any work requiring simply a long continued O. F., such as concentrating the silver-bearing lead from the fusion in the silver assay, roasting ores, or making crucible assays for lead, tin, etc., an arrangement like Fig. 52 will be very useful. The tube *a*, of the blowpipe, Fig. 1, carrying the platinum jet *b*, is inserted into a short tube *c*, Fig. 52, which passes through and is fastened in the movable metal block *d*, clamped at any height on the jointed rod *e* by the screw *g*. To the other end of *c* is fastened the rubber tube *k*, connecting with the vulcanized rubber regulator *R* and hand bellows *B*, which furnish the blast. A proper and easily arranged system of valves is provided in *B*. The metal (iron) foot of the stand should weigh about 530 grammes (18 to 19 ozs. avoird.).

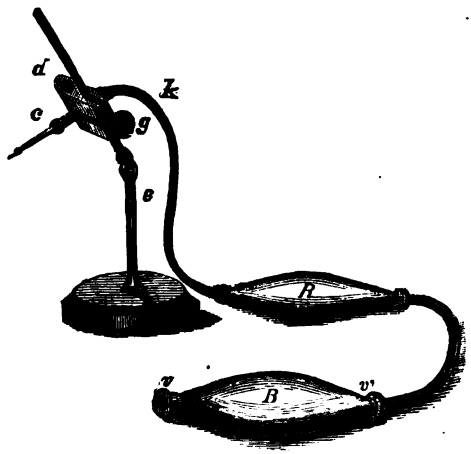

Fig. 52.

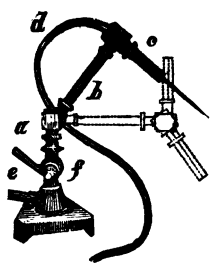

Fig. 53, *A*.

2. *Fletcher's hand-bellows* and *Herapath blowpipe.* The blowpipe, Fig. 53, *A* (about one-sixth the true size), consisting of the hollow, movable arms, *b*, *c*, attached to each other and to the stand *a*, by gas-tight joints, admitting of motion in only one plane, is the best general automatic arrangement for use with gas in quantitative blowpipe work

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to which such apparatus is adapted, as crucible assays, roasting and concentration of silver-lead, since the flame can point in any direction if the stand itself is also moved. The gas enters the system of tubes through the tube *e*, passing up through the hollow stand *a* and being controlled by the cock *f*; the blast, from the mouth or bellows, enters the end of the arm *c* through the rubber tube *d*. The whole is mounted on a heavy iron foot and when the arms *b* and *c* have about the proportionate lengths represented in the figure the apparatus is very convenient, the direction of the flame being under perfect control. Sometimes the arms *b* and *c* are made unnecessarily long for blowpipe work in assaying. The arrangement of the hollow arm *c* by which the flame is produced, is much the same as in Fig. 4, but the mouth of *c* is not narrowed at all. The crucible, when used, is heated in a charcoal, coke or fire-clay block, see 3, as for Hirschwald's apparatus, and the coal may rest on any suitable support, such as the wire triangle attached to the lamp, Fig. 2, or on any other support. The flame of the Herapath blowpipe can, by moving the arms, be easily caused to enter or remain outside of the furnace containing the crucible, the size of the flame remaining the same, while the evenness and good shape of the flame, render it very suitable for such work.

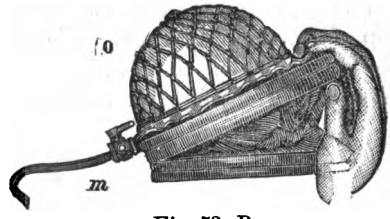

Fig. 53, *B*.

The hand-bellows, Fig. 53, *B* (about one-fourth the true size) is also well-adapted for blowpipe work, being compact and giving a steady blast without any additional regulator, since the elastic rubber sheet *o*, covered with a netting of cord, is itself the regulator. The blast issues through the rubber tube *m* and is controlled by a little cock. Both of these pieces of apparatus are made by the Buffalo Dental Mfg. Co., Buffalo, N. Y.

3. *Hirschwald's blowpipe furnace.* For the crucible assays for lead, tin and similar assays, Hirschwald's furnace (Plattner, *Löthrohrprobirkunst*, Leipsic, 1878, p. 574) is very convenient, although the burner itself is not so generally useful as that just described. The square furnace *g*, Fig. 54, and its cover *h*, made of graphite or pressed coke, like that used for galvanic batteries, will last during many operations. The furnace rests simply on a metal ring *e* at one end of an arm, on the other end of which is a smaller ring *f*, to hold hot crucibles. The whole upper part is carried by the rod *i*, revolving in the socket *d*. There is no side hole in *g*, but only one in the

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bottom, while otherwise the furnace is like the artificial square coal *F*, § 138, *d*, in size and shape. The arrangement of the burner is manifest from the section; the gas enters through *c*, while the air blast enters by the other rubber tube and issues from a fine jet at the top of the slender tube *a*. The rod *i*, turning in the socket *d*, with the sliding collars and binding screws, admit of adjusting the crucible in any position, but it is not so convenient to move the crucible, if

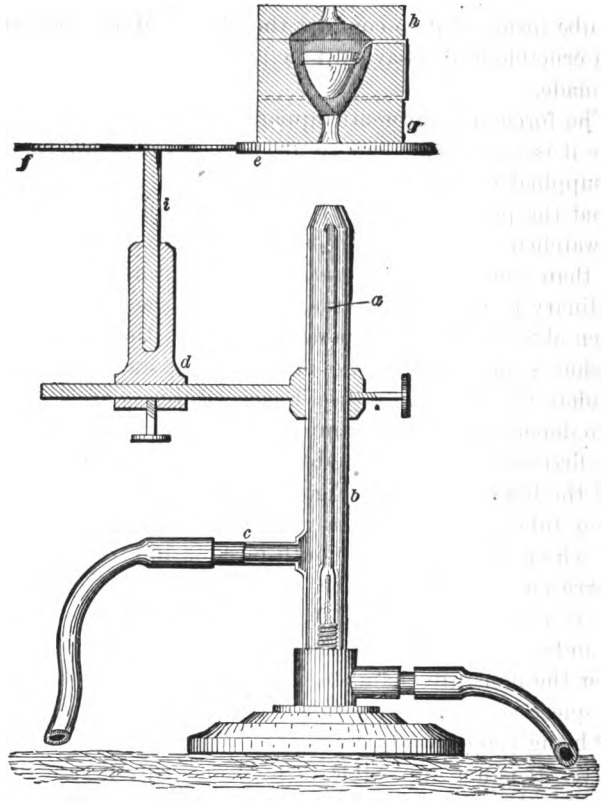

Fig. 54.

necessary during a fusion, as to move the flame itself, which can be done with the Herapath blowpipe. Fig. 54 shows how Hirschwald's burner can be constructed on the base of an ordinary Bunsen burner. Naturally, the ordinary artificial square coal already described can be used, or even a block of natural charcoal. Fletcher furnishes a little round furnace of fire-clay, with a cover of the same material, and dispenses with the wire ring inside of it, allowing the crucible to

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rest on three little projections on the inside wall of the clay furnace block. The arrangement is very serviceable.

It is perfectly feasible to employ simply the gas blowpipe, § 4, for all of this work, resting the square coal, with a hole in the bottom, on the triangle *E*, Fig. 2, and replacing the long metal tube of that blowpipe by a rubber tube, carrying the mouthpiece, as described in 4. Even when no gas is at hand an alcohol flame can be used, with the exercise of a little ingenuity in fitting up a cylindrical wick having a tube inside of it to conduct the blast. With such an arrangement a crucible lead assay by precipitation, § 170, can be very perfectly made.

4. The foregoing automatic apparatus has been described simply because it is very convenient to use for certain operations in laboratories supplied with gas, but not because it is at all necessary. It is true that the progress of the fusions in crucible operations can be easily watched and on that account better results are generally obtained than when the less experienced operator depends entirely on the ordinary forms of apparatus, but when the necessary experience has been obtained and the operator knows, without watching it, how the fusion is progressing, the ordinary apparatus is sufficient and the student should learn to use it successfully. Above all, he should learn to depend on the oil lamp, and in order that he may still be able at first to watch the crucible fusions without stopping the blast, even if the blowpipe stand, Fig. 52, is not at hand, he may replace the long tube *B*, Fig. 1, by a quite flexible rubber tube, into one end of which the trumpet mouth-piece is inserted, while the other end carries a short metal tube, to which the moisture-chamber *A*, Fig. 1, can be attached. (If it does not become too hot the moisture-chamber can be directly attached to the rubber tube.) The short tube, or the moisture-chamber, is held in the right hand, while the mouth-piece is kept in position by the left hand, the square-coal holder being properly adjusted before the oil lamp on any convenient support. It is not impossible to hold the trumpet mouth-piece by means of a short, flattened, horn or hard rubber tube inserted in it and held between the teeth, in which case the square-coal holder can be held in the left hand, but such an arrangement would not often be found advantageous, especially in assays requiring a very strong and long continued blast, like the tin assay.

B. Reagents.

148. *a. Test Lead.* The *granulated lead* should be fine enough to

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pass through the sieve, § 146, *d*. When necessary it is made by melting some pure lead, § 12, 9, pouring it into a well chalked wooden tray, gently rocking the tray to prevent solidification, and when the mass becomes sufficiently pasty throwing it into the air and letting it fall back into the tray. By continuing this the lead will be for the most part granulated. The fine part can then be sifted out and used. This is a better form of finely divided lead than the spongy lead obtained as in § 12, 9. Some *sheet lead* should also be provided, by fusing the finely divided, pure lead, if that form only is at hand. The test lead will also require to be tested for silver. Fuse one gramme of it in R. F. with borax on coal to a single button, and concentrate and cupel this, § 152. The resulting silver button is measured, § 154, *et seq.*, if large enough, and allowance always made for the silver when using the lead in assays for the precious metals.

b. Iron Filings, for the nickel and cobalt assay.

c. Sodium Chloride (*common salt*) fused, powdered fine and kept in a glass-stoppered bottle. It is used as a cover in crucible fusions.

d. Silver. Silver coin is dissolved in nitric acid, the solution diluted, filtered, treated with hydrochloric acid, so long as a precipitate of silver chloride falls, the precipitate is repeatedly washed by decantation with hot, distilled water containing a little nitric acid, finally brought on a filter and washed with hot water until the washings are no longer acid even after concentration; the silver chloride is dried and fused in a clay crucible with twice its weight of anhydrous sodium carbonate. The resulting silver button will be practically pure and can be hammered into thick ribbon for use. Small quantities of the silver chloride can also be reduced with soda B.B. on coal.

e. Borax Glass, powdered. It should be kept in a tightly closed bottle, and is used whenever borax glass is directed to be *mixed* with substances in assays.

f. Plattner's Flux. Mix very thoroughly ten parts by weight of freshly ignited and finely powdered sodium carbonate, thirteen of potassium carbonate, similarly prepared, five of powdered, freshly made borax glass and five of well dried starch powder, and keep the flux in a glass-stoppered bottle, since it will be useless if damp.

g. Arsenic. Heat powdered mispickel (arsenopyrite) in a closed tube, until the arsenic sulphide and a little arsenic have sublimed and then again heat the residue in another closed tube, saving the purer arsenic sublimate for use in the nickel and cobalt assays.

h. Graphite (*plumbago*). The best quality, used for moderately soft lead pencils, can often be purchased in rather large pieces, from

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which an exceedingly fine powder is to be gently scraped off for use in roasting ores. Lead pencils can be used if necessary. *Charcoal dust* is a substitute for graphite, but is not so good.

i. Soda-paper. Rather thin writing paper,* white and entirely free from any colored rulings, is drawn through a solution of one part by weight of crystallized sodium carbonate in two parts of water (or of one part of the anhydrous carbonate in 5.4 parts of water,) and is then dried slowly in a warm place. The soda must be quite free from sulphates. When dry the paper is cut into pieces 35 millim. long and 25 wide, and kept in a paper box.

j. Clay-Crucible Lining. Seven parts of very fine charcoal dust and one of elutriated fire-clay being weighed out, the latter is stirred with water until well mixed and then the coal dust is gradually worked in, the paste dried, powdered and preserved. To line a crucible, mix some of the above powder to a thin paste with water and gradually coat the crucible until the bottom lining is about 3 millim. thick, while it is made very thin above, as in Fig. 55. The lined crucible is smoothed inside with the brass stamp, Fig. 35, and dried over the alcohol flame.

Fig. 55.

k. Reddle. It is mixed to a thin paste with water and brushed over the inside of clay capsules, forming a very thin coating when dried at a moderate heat. This prevents substances from adhering to the clay capsule. Excess of reddle must be avoided. It is made, if necessary, by mixing finely divided, ignited sesquioxide of iron with about one half as much elutriated fire-clay or potter's clay. The iron oxide may be obtained by elutriating powdered hematite or strongly ignited limonite; also by strongly igniting ferrous sulphate on platinum foil, powdering it, with addition of a little ammonium carbonate, and again igniting it. This product does not require elutriation, since it can be rubbed to a very fine powder.

C. Preparation of Ores.

149. Very careful sampling is necessary. As in preparing ores for the furnace assay, a proper amount should first be passed through a sieve of 80 meshes to the linear inch, and from this stock several ivory spoonfuls are taken, here and there, this smaller sample ground very fine in the agate mortar and the final charge again taken in small quantities from different parts of this powder. Ores in which

* Thin filter paper should be used in qualitative work, since it yields much less ash.

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the free metal is in disseminated scales after grinding cannot ordinarily be well sampled for the blowpipe assays, but several assays of such will yield a good average result. Ores which can be entirely reduced to a powder that will pass through an 80 mesh sieve will, however, yield a true average amount of metal by one blowpipe assay, if carefully sampled as above. Gold ores alone may form an exception.

Ores which may have absorbed moisture should be dried thoroughly at a temperature of 100° Centigrade (212° Fahr.) before weighing. Substances that cannot be ground must be cut or filed very fine, unless a larger bit will furnish a fair sample. When great accuracy is necessary in such cases, several assays may be made and the results averaged.

II. THE SILVER ASSAY.

150. The blowpipe assay for silver corresponds very closely in theory to the scorification assay in the muffle and with moderately rich ores, from ten to fifty ounces per ton, which yield with the blowpipe buttons whose weight can be quite accurately ascertained by measurement, gives closely corresponding results. Richer ores give buttons requiring especial treatment when measured, see § 154, *et seq.*, or else the assays must be repeated several times, taking the average of their weight on the balance, since the closest weighing on the most delicate balances still leaves a possible error of several ounces, if only one assay is made on one centner of ore. No one would depend on blowpipe assays in buying and selling silver ores, but the prospector, provided with a moderately good balance and Plattner's ivory scale, will find that he can, after practice, come very near to the furnace assays.

A. Assay of Ores and Products which are not Alloys.

Preparing the Charge.

151. *a.* This method must be modified for *molybdenite* and for various easily reducible *oxidized lead compounds*, such as lead carbonate, litharge, etc.; see *d*, p. 190.

If the ores are very rich, average the results of three or four assays; if very poor, combine the three or four lead buttons from the concentrations of as many assays and make one fine-cupellation of the whole, after again concentrating, if need be. When several buttons are obtained from assays of the same ore, weigh them all at once.

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In preparing the charge weigh out one centner of the very finely powdered substance, pour it into the metal mixing capsule, § 146, *a*, using the brush only to remove the adherent dust, and add powdered borax glass and granulated test-lead. If the assay appears to contain much earthy matter, quartz, or oxides of iron, cobalt or tin, which make a rather infusible slag, take the ivory spoon heaped full of borax glass; otherwise only slightly heap the spoon. Too much borax glass tends to render the charge bulky and unmanageable, but enough must be added to render the slag sufficiently fusible. In case of necessity a little more can be added even during the fusion, but it is not advisable to interrupt that operation. Five measured centners, § 146, *e*, of test-lead, § 148, *a*, will generally suffice, but substances containing more than seven per cent. of copper or ten per cent of nickel or cobalt, will require more, as shown below:

Chalcocite (*copper glance*) with 80 % of copper requires 15 ctrs. of lead; *bornite* (*purple copper*) and *copper matts* with 50 to 70 % need 12 ctrs.; *gray copper* (*tetrahedrite* or *fahlerz*), *copper pyrites* and *copper matts* with 30 to 50 %, 10 ctrs.; *tin pyrites* and *bournonite*, 12 to 30 %, 7 ctrs.; lead *matts* containing over 10 % of nickel or cobalt, as well as *cobalt* and *nickel matts* and *speisses* generally, need at least 10 ctrs. of lead. A slight excess of lead does no harm, except by increasing the size of the charge and sometimes rendering it less manageable toward the close of the assay, when the charcoal has nearly burned away.

Thoroughly mix the whole charge with the handle of the ivory spoon, and pour it into a soda-paper cylinder, according to the directions in § 147, *a*.

Fusion for Silver-Lead.

b. Next bore a hole in the end (the cross-section, not in one of the longer, side faces) of a piece of sound charcoal, or far better, in a coal crucible, § 138, *b*, first boring the hole already there still deeper with the square borer, § 143, to about 7 millim., and then enlarging the upper part of the hole into a cavity with sloping sides, by cutting the coal away with the knife. This wider part should be about 12 millim. wide at the top, and may slope regularly down to the bottom of the hole, Fig. 56. If too much coal is removed the crucibles burn away too fast, while if the hole is too narrow the operation cannot be properly controlled and it will be found difficult to make the partly fused charge roll over as will be hereafter described. The packed soda-paper cylinder is firmly pressed into the crucible, with

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the folded part uppermost, and the crucible is placed in the clay crucible holder, the lower end of which rests on the end of the little finger of the left hand, while the cylinder itself is held by the other fingers and the left fore-arm rests lightly against the edge of the table. The lamp wick is raised so that the flame smokes slightly, and a moderate R. F. is directed upon the soda-paper, until the upper part has been thoroughly charred and the borax partly fused. If the lamp admits of it, the reservoir should be turned before beginning the fusion, so that the flame can be directed down upon the crucible, which must be slightly inclined, but not enough to spill the charge, Fig. 56. The flame must always be sufficiently reducing to prevent the oxidation of any of the lead, which now begins to appear in melted globules, but should still be oxidizing enough to allow the charred paper to burn slowly. When the lead globules begin to run together the whole charge must be covered with a good R. F.

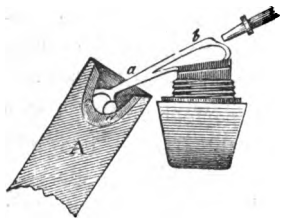

Fig. 56.

The fusion thus begun must be conducted with great care. Its object is to collect all of the reducible metals with the silver and the lead into *one* button, and partly to slag off, partly to volatilize other bodies. The upper part of the charge is easily fused, but to fuse the lower part it is necessary that the whole charge should *turn over and expose the paper at the bottom to the blast.* This cannot take place if the melted borax adheres to the coal, which it certainly will do if any lead is oxidized and dissolved in it. Should this happen the borax will begin to adhere to the coal and, if the trouble increases, the melted borax bead will lose its spherical form, adhere more and more widely to the coal, and finally spread out so as entirely to cover the charge below. To prevent this keep the charge (after the paper has been partly burned away at the top) and especially the already melted borax, entirely covered by a strong R. F., turn the crucible continually about its axis, and as the charge melts keep the R. F. directed mainly upon the melted borax, which will then generally remain quite free from the coal. Should it begin to adhere anywhere to the coal, preventing the free motion of the borax and lead from one part of the crucible to another, a strong R. F. must be immediately directed upon the borax at that spot and continued until the glass has separated from the coal and resumed its spherical shape. When the upper part is well fused and the slag is free from little lead globules the time has arrived for bringing the bottom of the charge

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and the unconsumed paper to the top. To do this incline the crucible holder more and blow a somewhat more pointed, but still reducing flame, upon the borax, so as to leave a space where the air can reach the lower part of the charge, turn the crucible holder from time to time and occasionally direct the flame toward the bottom of the charge, between it and the inside of the crucible, keeping a strong R. F. for the most part, however, upon the already melted slag, to prevent oxide of lead from being dissolved in it. In a short time the whole charge will roll over, bringing the paper and unmelted portion to the top, or at least to one side, when it must be at once covered with the R. F., which is thereafter to be kept steadily upon the borax glass, until the whole charge is in perfect fusion. The crucible holder is turned continually to bring the melted lead into contact with every part of the bead of slag, so that it may collect all of the reducible metals, and at the close of this operation not a globule of metal should be seen separate from the main lead button. In about two minutes after the paper has come to the top and burned away the first part of the operation, or the fusion proper, will be completed, the whole time occupying five or six minutes. Without stopping, the R. F. is changed to a moderately strong O. F., directed upon the melted lead, and this is the commencement of the *refining*.

A few more words are necessary about the fusion. A *hot flame* is as necessary as a *reducing* one, therefore the lamp flame must be large and the blast strong after the borax begins to melt. In a well conducted assay the borax will never adhere to the coal to any extent, but must tend always to a spherical shape and at the end of the fusion the borax slag must form a nearly spherical bead, partly covering the single lead button, while both are detached from and move freely around the crucible, Fig. 56. In general an assay in which the borax has dissolved much oxide of lead, spreading over the rest of the charge and adhering to the coal so that it cannot be restored to a proper condition by using a very strong R. F., as before directed, should be rejected; but if it must be saved at all hazards this can be done by very carefully cutting away most of the coal from it when cold, placing it with the coal uppermost in a new crucible, adding a little soda and fusing it in R. F. as before. The result will always, however, be doubtful.

c. The *Refining* which now follows is designed to volatilize sulphur, arsenic, antimony and zinc and to oxidize and slag off iron, cobalt, tin, etc. If there is much arsenide of nickel present it will not be readily oxidized, but may remain above the lead, from which

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it can be separated mechanically when cold, without loss of silver. Very soon after beginning to use the O. F. the lead commences to oxidize and the oxide dissolves in the borax, which now begins to spread over the coal. If rather impure the lead will remain in quiet fusion for a time, the O. F. being steadily directed upon it and the crucible inclined so that it may not run beneath the slag, which it has sometimes a great tendency to do, requiring a stronger blast to keep it fused and exposed to the air. Presently the lead will begin to move more rapidly, sometimes almost boiling, and the slag also bubbles briskly, showing that the refining is nearly or quite ended. The blast is then stopped, the crucible inclined slightly and the lead allowed to solidify. If it has a light, bluish lead-gray color and lustrous surface, often with a yellowish glaze of melted lead oxide over it, it is pure; if very dark and dull it contains antimony, sulphur or some copper. The two former can be removed by repeating the refining, while if the dark appearance is due to copper this cannot be thus removed, but the refining may then be considered complete after the fused lead has been in brisk agitation for at least a minute. The small lead globules which appear in and around the borax slag toward the end of the refining are due to the reduction of the recently oxidized lead by contact with the glowing coal and are disregarded, as they contain no silver. The refining occupies from half a minute to three minutes, or so, according to circumstances, and must be thorough, since impure lead is brittle and apt to sputter on the cupel. When cold the slag and lead are lifted out with the knife, carefully freed from coal and separated by hammering, after which the *silver-lead* is held in the brass forceps, § 146, *c*, and lightly beaten into a cube.

The student should practice the fusion and refining with easy ores at first, artificial mixtures containing fluor spar or other fusible earthy matters, with a little galena and oxide of silver, and afterward try the more infusible mixtures of blende, niccoliferous arsenides, very silicious ores, etc. If the crucible burns away before the refining is ended, the cooled charge can be readily transferred to another crucible.

d. The following cases require modifications of the foregoing process. *Molybdenite* is finely powdered and 1 ctr. is mixed with 1½ parts each of soda and borax glass and 5 of test lead, or more if much copper is present, and fused as before. When the molybdenite scales have disappeared the lead is refined with a strong O. F. to volatilize the molybdenum.

Oxidized lead compounds, if easily reducible and present in large

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quantity render the addition of test lead unnecessary, but soda must be added so as to quickly reduce the lead oxide, before it causes the borax glass to spread out and adhere to the coal. Five ctrs. (unless too rich in silver) are mixed with an ivory spoonful each of soda and borax glass, wrapped in soda paper and treated with a strong R. F., until all of the lead is reduced and collected in one button, leaving a spherical bead of slag, free from metallic globules. If necessary the R. F. is changed to O. F. and the lead refined as usual, but if it at once begins active ebullition under the O. F. it is pure enough.

Concentration for Rich Lead.

152. The silver lead is subjected to this operation, called also *scorification*, both because it is not easy to cupel much lead at once, so as to leave a pure silver button, and because the higher temperature required for cupellation would cause unnecessary loss of silver by volatilization.

A cupel of sifted bone-ash, § 12, 14, is struck, § 145, and its surface very strongly heated in O. F. to remove all moisture. The lead is placed upon it, the side to which any traces of charcoal dust may still adhere being uppermost, and is fused within the tip of the inner flame, which is then changed to a pure, not too strong O. F. The fused lead will begin to oxidize and the stand, which has been so far held in the hand, may now be placed on the table, the lamp having been previously fixed at the right height. The flame is directed rather across than upon the lead, Fig. 57, so that the oxide may accumulate on the farther side of the cupel. Never use any kind of R. F. after this, unless in case of *freezing*, as it must retard the oxidation. Should very much nickel be present it may form an infusible crust over the lead and then two to four ctrs. of test lead, in one or two pieces, must be added and the whole melted together with the inner flame before resuming the O. F. Should the lead cube have had any coal dust adhering to it this is burned away upon the cupel with a gentle O. F. before beginning the concentration. The operation is continued at as low a temperature as possible, until the lead has become very small, from half the size of a hemp seed to even less, according to the supposed richness of the ore. It is best to carry the concentration as far as possible, leaving enough lead to handle readily. The cupel is then slowly removed from the flame. Too high a temperature causes loss of silver; too low a temperature renders the lead liable to *freeze*, i. e., solidify,

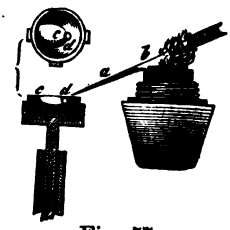

Fig. 57.

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and then the lead must again be fused in the inner flame before the oxidation can be resumed. A dark colored litharge shows copper, as pure oxide of lead is reddish-yellow. When the silver-lead is very rich and the concentration is carried so far that it contains about six times as much silver as lead, a grayish, friable mass may sprout from the solidified lead, consisting probably of suboxide of lead with silver, and this will cause a loss of silver, unless a small bit of test lead is fused with the mass in R. F. and the whole allowed to cool.

When cold the *rich lead* is lifted from the solid litharge with the brass forceps, or if this cannot be *very* easily done the litharge and lead are lifted from the cupel in one mass with the knife and then the litharge is separated from the lead by carefully pressing it from around the latter on the anvil, with the edge of the flat face of the hammer, cleansing the lead afterwards between the fingers. Care must be taken to leave no bone-ash on the lead ; a little litharge does no harm.

Cupellation.

153. This is called also the *fine-cupellation.* A cupel of washed or elutriated bone-ash, § 12, 14, is carefully struck, dried by intense ignition as before, and any cracks or rough spots removed by gently striking the bone-ash again. (The lower part of the cupel mould may be filled with sifted bone-ash, with a good facing of the washed ash.) The cupel must be at least so free from even the smallest cracks, after the ignition, as to afford sufficient clear space for the melted lead to move about in and to brighten out of contact with any of them. The rich lead is then placed on the cupel, rather toward the border than the centre, the cupel stand is held with its foot resting on the left little finger and the lamp raised so that the flame can be easily directed anywhere upon the cupel, while the latter is easily moved and turned with the hand. The cupel mould being slightly inclined, so as to prevent the lead from rolling at once from the outer border to the centre, the button is fused with a gentle O. F. and the cupel then brought nearly vertical, with the O. F. directed on the bone-ash just before the lead, rather than on the lead itself, Fig. 58, so that the ash may be hot enough to at once absorb the fused litharge, none of which must remain on the surface of the cupel. By properly directing the flame and turning the stand the button is slowly driven about, particularly toward the end of the operation. The end is shown, when there is any considerable amount of silver present, by a play of colors on its melted surface, due to a

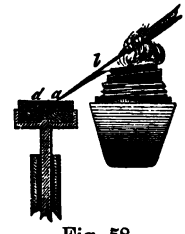

Fig. 58.

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thin film of litharge. After this has lasted a few seconds the button, previously red-hot but not very lustrous, will suddenly become *bright and fixed on the cupel*, and this *brightening* must always be accomplished on the clean portions of the cupel, the button being nearly touched at the last with the point of the inner flame, to remove the last trace of litharge. In very small buttons the play of color is invisible, but they also brighten and become fixed, in a marked way. The button can, indeed, be made to move afterwards by a stronger blast, but not by the moderate blast used for the cupellation.

After brightening the silver button is slowly removed from the flame and examined with the lens, to detect any yellowish film of litharge, pure silver being silver-white and lustrous. A considerable amount of gold may make it yellow, but the appearance is not the same as when litharge covers the silver. If litharge is present it is removed by fusing the button a very short time in O. F.

Large buttons are apt to *sprout*, or throw out branch-like projections, if rapidly cooled, and thus cause loss by spirting. If heated strongly after brightening the button loses silver, which may combine with the lead oxide and form a rose-colored coating on the cupel, § 83, *a*, different from the bright orange-red coating often formed by the litharge alone near the rim of the cupel. A large silver button exposed to the O. F. after brightening sometimes shows dull spots, probably of silver and its oxide, which may be removed by a momentary treatment with the inner flame. A dark stain on the cupel indicates copper, which may also make the silver appear dark and may render it flatter than a pure silver button. In this case a centner or less of lead in one piece, according to the apparent amount of copper present, is fused with the button in the inner flame, before detaching it, and the cupellation repeated. This is a better plan than to add all the lead necessary to separate much copper to the charge for fusion for silver-lead.

Sometimes when nearly bright the silver button will adhere to some projection on the cupel from which it cannot be detached during the cupellation, nor properly after it. A bit of lead must then be fused with the button, which can afterward usually be forced away by a proper blast and by inclining the cupel. Should the button be *drowned* or surrounded by the accumulation of litharge not absorbed by the cupel, through too feeble a blast, the blast is stopped, the button removed, after fusing a bit of lead with it, if it is too small to be handled with the forceps, and the cupellation finished on a fresh cupel. A very slight accumulation of litharge surrounding the brightened button can, however, be made to sink into the bone-ash

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by using a strong O. F., holding the cupel at a distance. Automatic apparatus is not suitable for this cupellation.

Determination of the Weight of Silver Buttons.

Weighing the Silver Button.

154. The blowpipe assay yields as much of the silver in an ore as any furnace assay, and may even yield more, and the sampling of most ores can be perfectly made, so that the only obstacle to a correct estimation of values by it lies in the difficulty of exactly determining the weight of such small silver buttons. Balances are made whose index needles deviate one division on their scales for $\frac{1}{2}\frac{1}{0}$ milligr. and by averaging the readings of several vibrations such balances can be made to indicate $\frac{1}{5}\frac{1}{0}$ milligr. If one assay is made on one ctr. of ore then each milligr. of silver will represent 291.66 ozs. of silver per ton of ore and $291.66 \div 50 = 5.83$, the possible error in weighing. By making several assays and weighing all the buttons together the error can safely be reduced to reasonable limits. A good balance whose index needle deviates only one division for $\frac{1}{1}\frac{1}{0}$ milligr. can be used to indicate $\frac{1}{2}\frac{1}{5}$ milligr. and for ores of 100 ozs. or more the above balances will be found to give very good results on the average of several assays. For poorer ores it will, however, be found more convenient and accurate to measure the buttons, as will be presently described, unless a large number of duplicate assays are made.

When the button is large enough to weigh it is raised from the bone-ash with the points of the button pliers, § 144, *c*, the bone-ash brushed from its lower surface with the button-brush, the button examined with the lens and weighed if clean. Should it adhere very firmly to the cupel and appear dark either above or below, this is probably due to copper and the button should be re-cupelled with a little test-lead.

Measuring the Silver Button.*

155. An ore containing 50 ozs. of silver per ton would yield by assaying one ctr. a silver button weighing only 0.17 milligr., while one with but 10 ozs. would yield a button of only 0.0343 milligr., necessitating many assays before a weighable amount of silver was obtained.

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* If the button contains gold its weight cannot be accurately obtained by any modifications of the measuring process, but one per cent. of gold will scarcely cause an appreciable error.

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Plattner's scale, § 137, furnishes a ready means of approximately determining the weight of such buttons, but it does not answer well for buttons coming above the 19th transverse line, corresponding to about 55 ozs. per ton. When a fair degree of accuracy is desired it is better to divide the silver-lead from richer ores into a suitable number of pieces with a sharp, smooth-edged knife, and concentrate and cupel the pieces separately, so as to bring the silver buttons within the above limits.

When the button is to be measured it is securely, but gently seized with the small, pointed forceps, § 144, *d*, being loosened from the bone-ash, if necessary, with the point of the knife. If it adheres too firmly to the bone-ash it must not be forced off, but the bone-ash immediately beneath it is lifted off, with the adhering button, and the two are placed on smooth paper and gently rubbed with another bit of paper under the finger. By such treatment the button should be readily freed from the bone-ash. If this is not the case it is certain that the silver is not pure and the button should be recupelled with a very small bit of lead. It is absolutely necessary that the silver should be pure, because otherwise it will never assume so spherical a shape as it should after the cupellation, and, being at least too much flattened, if not quite irregular in shape, will almost certainly give too high results by measurement, if it admits of accurate measurement at all. Nearly all errors in blowpipe assays of silver ores when the buttons are measured result in yielding too high a value. It will not do to remelt the button on coal, in order to make it spherical, since the formulas used in the calculations are based on the beads obtained by cupellation, which are always somewhat flattened.

Much care and delicacy of touch are required to avoid injuring or losing these minute buttons, but they can be handled even when obtained from ores with less than 6 ozs. of silver. It is better, however, to unite the rich-lead from several assays of very poor ores and concentrate this to a size suitable for the fine-cupellation, since buttons from ores of less than 10 ozs. or so are very hard to handle.

When ready for measurement the button is placed upon the ivory scale and moved along it until the two convergent lines are exactly tangent to its sides, when the weight of the button in milligrammes is read off from the right hand row of figures. The best way to attain skill in using the scale is not to use large weighed buttons, but to practice with the minute buttons from blowpipe assays of ores whose values have been accurately ascertained by careful furnace assays. With practice, the results on ores ranging from 15 to 40 ozs. will not

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vary more than one or two ounces from the right result. For ores of over 55 ozs., at most, it is better to divide the silver lead into a suitable number of nearly equal pieces and cupel these separately, because the larger silver buttons are more liable to flatten irregularly, while their value also rises very rapidly as their diameters increase, thus making close results by measurement almost impossible.

As already stated, the author can recommend only Lingke's own ivory scales as always accurate. When a balance weighing even to only ½ milligr. or 1 milligr. is used for weighing out the charge of silver ore the ivory scale furnishes a very valuable instrument for prospecting work.

156. The use of the scale requires practice, and when this is wanting other means of measurement may be employed, affording equal accuracy, but hardly more. Their only advantage is that they are more certain in unpracticed hands.

Two such means will be described briefly; the microscope and Rueger's measuring apparatus or button micrometer.

A microscope with a magnifying power of about one hundred diameters and provided with an eye-piece micrometer whose divisions are 200 to the inch answers well. If the value in inches of the micrometer divisions, for the particular magnifying power used, cannot be obtained from a reliable manufacturer and no reliable stage micrometer is at hand, by which they might be measured, their value can still be reckoned by means of perfectly round silver buttons, obtained by blowpipe assays of one ctr. of ores of known value, containing 20 to 40 ozs. of silver, using for the calculation the formula, p. 198, and remembering that one inch equals 25.399 millim. The weights of silver buttons measured by the microscope are also to be calculated by this formula, which the author has found to give results within two ounces of the furnace assay on two ores containing respectively 12 and 40 ozs.

The microscope will be found more convenient than Rueger's apparatus, because by simply revolving the micrometer eye-piece several measurements of a button can be quickly taken, when its slightly irregular shape necessitates this. The cost would also be no more.

If the button is too irregular, various measurements differing more than one-twentieth from each other, it should be recupelled with a very little lead. As in the case of the ivory scale, too large buttons should not be measured; it is better to divide them, or repeat the assays and weigh the silver. The usual error is in bringing out too

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much silver and the button should be recupelled unless it is perfectly bright and smooth.*

157. Rueger has invented a measuring apparatus depending on the use of the micrometer screw. This apparatus is shown in Figs. 59 60; the latter representing a section through *C D*, Fig. 59.

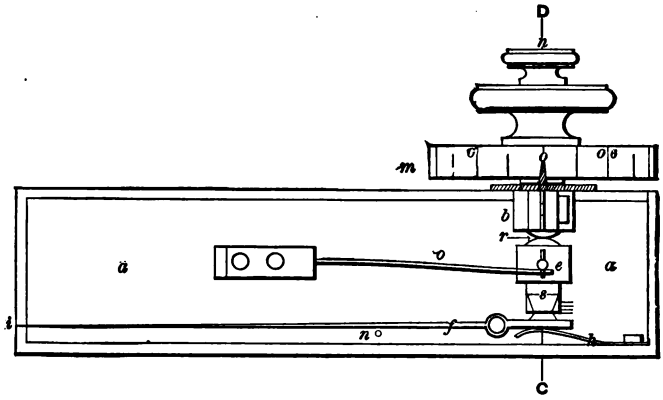

Fig. 59.

After loosening the screw *n* the graduated circle *m* is turned until the index *o* points to zero on the circle, at the same time that the index arm *f* coincides with the mark *i*, the latter condition being secured by the use of a lens. The screw *n* is then clamped and the button to be measured is placed on the plate *a*, between the jaws of the instrument, at the dotted line, by *f*, Fig. 60, exactly upon a line drawn on the plate *a* as a guide, the milled head having been turned so as to open the jaws. The milled head is again turned so as to bring the index arm *i* and the line into coincidence once more, when the jaw *s* will have moved over a less distance to produce this result than it did before the button was placed between the jaws. The distance is read off, partly by means of the short parallel lines, to the right of *s*, Fig. 59, the distance between two of which represents two revolutions of the micrometer screw, and partly by the graduated circle

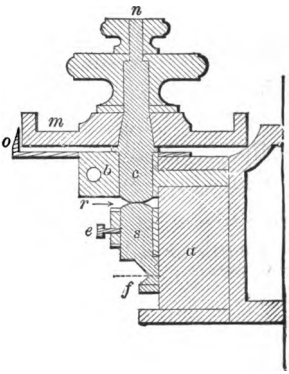

Fig. 60.

* With exceedingly poor ores, one or two ounces per ton, a fairly approximate measurement can be made by at once putting the cupel, with the button still on it, under the microscope.

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m, on which the fractions of a revolution are read. Rueger's instrument is made so that the jaw *s* advances one-fifth of a millimeter for each revolution of the circle *m* and Hüenich* has given the following formula for use with it in obtaining the weight of silver buttons by measurement.

$D^3 \times 5.5622$ = the weight of the button in milligrammes, D being its diameter in millimetres. If the measurement is made in inches the student should remember that 1 inch = 25.399 millim.

Rueger's instrument is compact and small, but is not so convenient otherwise as the microscope, because with the latter a little adhering bone-ash is not so likely to cause errors and because it is easier to take readings of different diameters of the buttons with the microscope.

Cupellation Loss.

158. Plattner has determined the amount of silver lost during cupellation for all proportions of silver in any substance, down to one per cent., when treated with different quantities of lead. The results are given in the following table :

Table Showing the Loss which occurs in Cupelling Silver with various amounts of Test Lead.

Weight of the silver button obtained by cupellation.	16 ctrs. lead.	13 ctrs. lead.	11 ctrs. lead.	8 ctrs. lead.	6 ctrs. lead.	5 ctrs.	4 ctrs.	3 ctrs.	2 ctrs.	1 ctr.
	The cupellation loss suffered by the silver present in the assay is :									
Milligrammes.	Milli.	Milli.	Milli.	Milli.	Milli.	Milli.	Milli.	Milli.	Milli.	Milli.
99.5 to 99.75	—	—	—	—	—	0.50	0.45	0.39	0.32	0.25
90.	—	—	—	0.83	0.69	0.47	0.42	0.36	0.29	0.22
80.	—	—	—	0.75	0.64	0.44	0.39	0.33	0.26	0.20
70.	—	—	0.82	0.68	0.58	0.40	0 35	0.29	0.23	0 18
60.	—	—	0.74	0.61	0.52	0.36	0.30	0.:6	0.20	0.16
50.	—	—	0.65	0.54	0.46	0.32	0.26	0 23	0.17	0.14
40.	—	0.62	0.55	0.46	0 39	0.27	0.22	0.20	0.15	0.12
35.	—	0.57	0.50	0.42	0.36	0.25	0.20	0.18	0.13	0.11
30.	—	0.51	0.45	0.38	0 32	0.22	0.18	0.16	0.12	0.10
25.	—	0.45	0 40	0.34	0.29	0.20	0.16	0.14	0 10	
20.	0.45	0.39	0.35	0.29	0.25	0.17	0.14	0.12		etc.
15.	0.37	0.32	0.28	0.23	0.20	0.15	0.12	0.10	etc.	
12.	0.32	0.26	0 23	0.19	0.17	0.13	0.11			
10.	0.27	0.23	0 20	0.17	0.15	0.11	0.10	etc.		
9.	0.25	0.21	0.18	0.16	0.14	0.10				
8.	0.22	0.18	0.16	0.15	0.13	0.09	etc.			
7.	0 20	0.16	0.14	0 13	0.12	0 08				
6.	0 17	0.14	0.12	0.11	0.10	0 07				
5.	0.14	0 12	0.11	0 10	0 09	0 06				
4.	0 11	0 10	0.09	0 08	0.07	0 05				
3.	0.09	0.08	0.07	0.06	0 05	0 04				
2.	0.07	0.06	0 05	0.04	0 04	0 03				
1.	0 05	0 04	0 04	0.03	0.03	0 02				

* The details of the calculations on which this formula and the graduation of Plattner's ivory scale are based are fully given in Plattner's *Löthrohrprobirkunst*, Leipsic, 1878.

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Beginners increase the loss by cupelling too hot. The total amount of lead used, from the charge for fusion to the end of the cupellation, is taken, as well as any lead or bismuth in the ore, although this is unusual except for the greatest accuracy, while no allowance should be made for the cupellation loss in commercial assays or with measured silver buttons. When the balance will indicate 0.05 milligr. the loss is calculated to two decimal places, being reckoned by difference for buttons between 50 and 60 milligr., or between 60 and 70, etc.

B. Alloys.

159. These may contain chiefly:

a. Silver, or *silver and gold.* File the alloy clean, weigh a fragment of 80 to 100 milligr., place it in a coal crucible properly bored out, and add 1 ctr. of test lead and a half-spoonful of borax glass, increasing the lead to 5 ctrs. according to the copper present. Fuse in R. F. until thoroughly alloyed with the lead, refining afterward if necessary, § 151, *c*, and cupel the cleansed lead button at once, only concentrating, § 152, when too much lead is present.

b. Copper or *nickel and silver* (in presence also of *tin* see *e*). Weigh, either in filings or clippings, one-half ctr., mix it with 10 ctrs. of lead and one spoonful of borax glass, wrap in soda paper, § 151, *a*, fuse in a strong R. F., until all reducible metals are collected in one button, concentrate and cupel as usual, making a second cupellation with ¼ to ½ ctr. of lead if much remaining copper renders this necessary, § 153. *Brass* requires a heaping spoonful of borax glass and the fusion should be finished by directing the R. F. only on the borax glass, to volatilize all the zinc, strongly heating the lead a short time after it appears pure.

c. Lead or *bismuth and silver.* Two to ten ctrs. of the *lead* in bits are fused for a short time with O. F. in a coal crucible with an even spoonful of borax glass, until pure, § 151, *c*, and the button is then concentrated and cupelled, observing the remarks in § 153, in presence of much copper. Quite pure lead need not be refined.

Bismuth is pulverized and 5 ctrs. fused with borax glass in a cavity in the cross section of a sound piece of coal in O. F. for a short time and carefully poured out upon the anvil. It is concentrated like lead, remembering that it is brittle, and the rich bismuth is cupelled with addition of one piece of lead weighing 30 to 40 milligr.

d. Tellurium, antimony or *zinc and silver.* (For *brass* see *b.*) Treat 1 ctr. with 5 ctrs. of lead and one spoonful of borax glass in R. F., until all the metals are collected into one button and then

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refine and finish as usual. Tellurium requires repeated concentration (more properly *scorification*) with 5 ctrs. of lead each time, until the rich lead button shows a bright lead-gray surface, when it is cupelled. Should the silver button still contain tellurium and have a reticulated, grayish-white, dull surface, it is again cupelled with 1 ctr. of lead. The cupellation loss must be here added.

e. Tin and silver (even if the former is in rather small proportion). Charge the finely divided alloy with 5 to 15 ctrs. of lead, according to the copper present, ½ ctr. of soda and an even spoonful of borax glass, wrap in soda paper and fuse with a strong R. F., until all the metals are collected in one button, which is then treated with the blue, inner flame, so that the easily oxidizable tin may slowly be taken up by the melted glass. As soon as globules of reduced tin appear at the edges of the glass the lead is cooled, transferred to another coal, with a spoonful of borax glass, and fused first with R. F. and then oxidized as before, until it shows a clean surface, free from oxide of tin. It is further treated as in § 153.

f. Mercury and silver. One ctr. or so is weighed in the balance, covering the pan or metal capsule with glazed paper, and is then very slowly distilled in a glass tube, Fig. 61, until the residue has been red-hot for some time. If considerable the residue is shaken out of the tube, to which it should not have been made to adhere by too great heat, and is treated in R. F. with 1 to 3 ctrs. of lead, according to the copper present, and one-half spoonful of borax glass and then cupelled as usual. When the residue cannot be shaken out the bulb is cut off, filled with one ctr. of lead and one-half spoonful of soda, the whole fused in R. F., the silver-lead button separated from the glass when cold and cupelled.

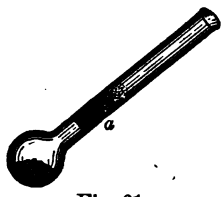

Fig. 61.

g. Iron or *steel and silver.* Divide the substance finely by filing or cutting (first softening steel by heating) into pieces not weighing more than 30 milligr. and mix 1 ctr. with ½ ctr. powdered sulphur, 8 ctrs. lead and one spoonful of borax glass, transfer to soda-paper and fuse to a globule in R. F. A heaping spoonful of borax glass is then melted with the globule and the whole treated with a strong O. F., directing this upon the lead as soon as it emerges from the slag, and holding the crucible so as to favor this emergence. When sufficiently refined and bright, the lead is cupelled.

III.—THE GOLD ASSAY.

160. The great value of gold and the very small quantities usually

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present in ores render the blowpipe assay, except for quite rich ores, tedious, while it is but approximately correct in any case. By careful measurements, with the ivory scale or otherwise, fair comparative results may, however, be obtained from moderately rich ores, since the assay yields as large a proportion of gold as the furnace assay. Gold and silver, if occurring together, are found together after cupellation.

A.—Ores and Products not Alloys.

161. *a. Gold ores and products with little or no silver.* The ore is charged and the assay made as directed for silver, §§ 151 to 153. Should the resulting button have a pure, deep gold color it is weighed or measured. As little as 2 per cent. of silver will give gold a brass-yellow color, and should the light color indicate any silver it is separated as in § 162, *a*.

b. Substances containing more silver than gold. Assay one ctr. as usual and from the weight and color of the auriferous silver button obtained judge how many such buttons will yield enough gold to be quantitatively determined, remembering that the aggregate weight of the auriferous silver buttons must be large enough to be determined by the balance. Should the first button, for instance, weigh less than 0.1 milligr. and appear to contain little gold, as many as 20 assays of one centner each might have to be made, and fewer in proportion as the color indicates more gold, this metal deciding the number to be made. Should there be two or three per cent. of silver and this, too, apparently quite auriferous, two or three assays may suffice. They are made just as before, allowing more lead according as more copper is present, and concentrating the auriferous silver-lead cubes in twos or threes, unless they together exceed 15 ctrs. in weight. The resulting rich lead buttons are all concentrated at once to one rich lead button, the smallness of which depends on the silver and gold present, and this is cupelled fine, with addition of lead if necessary. The auriferous silver is then separated.

c. Iron and copper pyrites, or *ores and products containing much metallic sulphides,* may be concentrated as follows: The fine powder is roasted in charges of 3 ctrs. in clay capsules coated with reddle, just as directed for copper ores containing sulphur, until all the sulphur is oxidized. The whole charge, 12 to 20 ctrs. according to the richness of the ore, may be roasted at once in a thin porcelain dish, or an iron dish coated with reddle or fire-clay and water, over a powerful flame (an alcohol lamp with double draught, or a compound gas burner), keeping the charge at a low red heat and stirring it oc-

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casionally, until no more sulphurous fumes escape. The roasted ore is then gently heated in a porcelain dish, covered with a watch glass, with enough hydrochloric acid to dissolve the oxides of iron, copper, etc. Some chloride of silver dissolves, but no gold, unless some oxide like the higher oxides of manganese is present, which would evolve chlorine with the acid. Earthy gangues may also remain undissolved. When all the soluble part is dissolved the whole is evaporated to dryness on a water bath and treated with enough water to dissolve the soluble chlorides, and then with some solution of ferrous sulphate, to precipitate any gold still in solution, after which the whole is stirred and allowed to settle. When clear it is filtered, the residue, which will contain the now insoluble chloride of silver and the metallic gold, is washed on the filter with hot water and dried on it. Five ctrs. of lead and a spoonful of borax glass is mixed with the powder, the whole wrapped up in the filter, after cutting off the clean paper, and treated with a gentle O. F. in a coal crucible until the paper is nearly consumed, when the metals are collected into one button under a strong R. F. and this is cupelled for auriferous silver, which is then treated as in § 162, *a*. All of the silver in the ore is thus obtained.

B. Alloys.

162. *a. Gold and silver.* When an alloy of gold and silver containing not more than one part of gold to two and one-half of silver is heated with moderately strong nitric acid all of the silver dissolves while the gold is left as a dark residue, but if more gold is present some silver must be added. The operation is called *parting.* (By comparing the streak of the alloy on a touchstone with the streaks of a series of alloys of known composition an opinion of its composition can be obtained.) Two per cent. of silver gives to gold a light brass-yellow color, and sixty per cent. conceals the yellow color entirely. Should the alloy have a brass-yellow color add two and one-half times its weight of pure silver; if a very light yellow, twice its weight; a silver-white alloy requires about one-half its weight, to make certain of securing a correct result, with unknown ores, although the alloy from auriferous silver ores and products in fact rarely needs any additional silver.

From 50 to 80 milligr. of the alloy being weighed out, it is fused with the necessary amount of pure silver on coal, in a shallow cavity, with a little borax glass, using a moderate R. F. and keeping the flame upon the glass, until the well fused metals are very thoroughly mixed. If the alloy contains other metals and the silver is also to be

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determined, the weighed alloy is fused with 1 to 2 ctrs. of lead, in one piece, and some borax glass in R. F. on coal, cupelled and the argentiferous gold button weighed before adding the pure silver. To determine the cupellation loss reduce the litharge and the bone-ash containing it with soda and borax glass on coal, cupel the lead thus obtained and add the weight of this silver to that already found as below. Unless very small the button, now having the proper composition, is hammered thin between paper, annealed by heating to low redness on coal in O. F., and gently heated in a test-tube with a little nitric acid. Only moderately strong nitric acid should be used at first, of 1.16 specific gravity, since this attacks the alloy less violently and tends to leave the gold in one mass. The silver is rapidly dissolved out, while the gold remains as a dark brown or blackish, spongy mass, or in separate particles. When the evolution of bubbles ceases, indicating that the silver has been for the most part dissolved, the acid is boiled for a short time, the solution poured off into a porcelain dish and the gold in the test-tube again boiled for some minutes with fresh acid, this time using stronger acid, sp. gr. 1.26, to dissolve the last traces of silver. The acid is poured off and the gold boiled two or three times, with fresh portions of distilled water each time, to remove all nitrate of silver, the water being decanted into a white porcelain dish, in order that any particles of gold accidentally carried over may easily be seen. The gold will, however, remain in the test-tube if the operation is carefully performed.

The test-tube is then filled quite to the top with cold water, a clean porcelain crucible placed over its mouth, bottom side uppermost, and the two quickly inverted, after which they are placed together in a porcelain dish, the mouth of the test-tube still resting on the bottom of the crucible, which stands in the dish. The water will remain in the tube and the gold quickly settle to the bottom of the crucible. When it has done so the tube is carefully raised, until the water flowing from it has filled the crucible, after which the tube is slipped over the brim of the crucible by a dexterous motion, leaving the gold in the crucible, which is now also full of water. The water is poured off, any drops remaining are carefully soaked up with filter paper and the gold cautiously dried at a gentle heat, after which the crucible is somewhat strongly heated to render the gold more compact. During this heating the gold assumes a dull, yellow hue, but will not adhere to the crucible, if this is not too strongly heated. The gold is now detached from the crucible very carefully with a smooth, pointed piece of wood and weighed.

If the alloy contained more than traces of tin or antimony, the

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gold should first be mixed with one centner of lead and a little borax glass in the mixing capsule, transferred to a soda-paper cylinder, fused in R. F. on coal as in an ordinary assay of ores, and the resulting lead button cleansed from slag by hammering and cupelled. The lead will be brittle if much gold is present and must be gently hammered. The cupellation yields pure gold.

b. Gold, silver and copper. Fuse 30 to 50 milligr. of the alloy with some borax glass and 3½ to 8 ctrs. of lead, according to the copper present, in a strong R. F. on coal, and concentrate and cupel as usual, following the directions in § 153 if the cupelled button still contains copper, and finally separating the gold and silver as in *a*.

c. Gold and platinum. 1. If the alloy contains much platinum dissolve 30 to 40 milligr. by heating with aqua regia, add to the solution sufficient solution of ammonium chloride, evaporate to dryness at a temperature slightly below that of boiling water, and wash the residue on a little filter with alcohol of about 80 per cent. until the alcohol is no longer colored yellow as it drains through. The gold can be precipitated by heating the filtrate, after water has been added and the alcohol expelled by evaporation, with ferrous sulphate. When it has completely settled it is collected on a filter, washed, dried, rolled up and burned over the agate mortar. The charred residue is carefully mixed with about 50 milligr. of borax glass, wrapped in a soda paper cylinder and the gold collected into one button by treatment in O. F. on coal, while the filter ash is slagged off. The button is cleansed by hammering between paper and weighed.

2. If there is but little platinum in the alloy fuse 30 to 50 milligr. of it with three times the weight of pure silver, as in *a*, beat the button as thin as possible, anneal it, bend it into a roll and boil it two or three times with strong nitric acid, rather longer than for a simple alloy of gold and silver. The platinum and silver will dissolve, leaving the gold behind. It is well washed by boiling with water, dried, ignited gently in a porcelain crucible or on platinum, and weighed. Should the platinum originally have amounted to several per cent. of the alloy, this gold may still contain a little of it and will not then show a pure gold color. In this case the whole operation of fusing with silver, etc., is repeated.

d. Gold, platinum and silver. Unless the amount of silver is to be determined follow *c*; otherwise the silver is first separated by means of sulphuric acid. At least one and one-fourth part of silver and not more than two parts must be present for every part of gold and platinum; with more silver some platinum seems to be dis-

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solved. Thirty to fifty milligr. of the alloy being weighed out and fused, if necessary, with enough pure gold or silver beside borax on coal to secure the necessary proportion of silver, it is beaten very thin, annealed, rolled up, weighed to avoid risk of error by mechanical loss, and boiled in a porcelain vessel with strong sulphuric acid. After boiling for ten minutes the acid is allowed to cool, poured off and the residue boiled for five minutes with fresh acid. The silver dissolves as sulphate, leaving the gold and platinum in the little roll, which is boiled with water, dried, ignited and weighed. After this the platinum is separated as in *c*.

e. Gold, platinum, silver and copper. The copper is removed by cupellation with lead, as in *b*. When much platinum is present the lead cannot sometimes be entirely separated on the cupel and in this case the remainder of it is removed by treatment with boric acid. A shallow cavity is bored in a piece of coal or in a coal capsule, § 138, *c*, the alloy placed in it beside a little previously fused boric acid, and the whole treated with the tip of the inner flame. As soon as the button melts the coal is inclined so that it may emerge from beneath the boric acid and be largely exposed to the air, while still remaining in contact with the acid. The tip of the inner flame is now directed without interruption upon the glass, until all of the lead has been oxidized and dissolved in the glass, leaving the button lustrous and pure, provided all copper had been separated during the cupellation. The alloy is then treated as in *d*.

f. Gold containing iridium. The alloy is treated with aqua regia, which dissolves the gold and leaves the iridium as a black powder, which can be filtered out from the diluted solution and washed. The gold is precipitated from the filtrate by ferrous sulphate and further treated as in *c*. Any copper present must first be separated by cupellation with three to five parts of test lead and the cupelled alloy freed from traces of lead as in *e*, if necessary.

g. Gold containing palladium. Follow *c*, 2, since palladium behaves in this case like platinum. In presence of metals which oxidize on ignition in air these must be removed by cupellation as in *e*, followed by treatment with boric acid, if necessary.

h. Gold containing rhodium. If the gold predominates the alloy will dissolve entirely in aqua regia and the gold can be thrown down by ferrous sulphate. Rhodium can be separated from finely divided alloys with platinum, iridium, osmium and gold, by fusion with potassium bisulphate, which only attacks the rhodium. Thirty to fifty milligr. of the alloy having been fused beside borax on coal with three times the weight of silver, the button is further treated as in

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c, 2, with nitric acid. The remaining roll contains the gold and rhodium and it is well washed, dried and fused over the alcohol flame in a platinum vessel with potassium bisulphate, the heat being gradually raised to low redness. Sulphurous oxide escapes and the rhodium dissolves to a dark-red or black salt, together with any residue of silver. When the evolution of sulphurous oxide ceases and the red-hot, fluid salt is quiet, it is poured off from the gold upon the anvil, the last portions being removed as far as possible with the steel spatula. The fusion is repeated with fresh potassium bisulphate, which is also poured off, and the gold roll is then boiled a few times with water, dried, ignited and weighed. By once repeating the whole process, of fusion with silver, etc., the last traces of rhodium can be removed, and this should be done with alloys containing much rhodium.

i. Gold and mercury. Distill 50 milligr. or so of the amalgam as in § 159, *f*, and cupel the residue with one ctr. of lead, separating any silver by *a*. When the residue is exceedingly small put more amalgam into the tube and distill, repeating this until a sufficient residue is obtained.

Weighing or Measuring the Gold Button.

163. If there is enough of it the gold may be weighed just as it comes from the *parting*, but it must be obtained in a button when measurement is necessary. Since the blowpipe assay can yield weighable amounts of gold from even moderately rich ores only by tedious repetitions, it will generally be found advantageous to measure the gold.* For this purpose, if not already obtained as a button by previous cupellation, the spongy gold is wrapped in as little, very thin sheet lead as possible, free from gold and silver, and fused beside borax glass in a cavity on coal in R. F., until the lead has alloyed with the gold in one button. This is freed from slag by gentle hammering, avoiding rough handling if the lead is brittle from presence of much gold, and is then cupelled. The resulting gold button is then measured, on the ivory scale, or otherwise. The following table shows the weight of gold buttons corresponding to the various transverse lines, as indicated by the left hand figures of the scale.

* If silver is to be determined the button must be *weighed* before the parting; alloys cannot be accurately valued by measurement.

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TABLE FOR USE WITH PLATTNER'S SCALE.

No. of the Transverse Line.	Percentage of Gold.	No. of the Transverse Line.	Percentage of Gold.
26.	1.065	13.	0.133
25.	0.946	12.	0.105
24.	0.838	11.	0.081
23.	0.737	10.	0.061
22.	0.645	9.	0.044
21.	0.561	8.	0.031
20.	0.485	7.	0.021
19.	0.416	6.	0.013
18.	0.353	5.	0.008
17.	0.298	4.	0.0039
16.	0.248	3.	0.0016
15.	0.205	2.	0.0005
14.	0.166	1.	0.00006

Thirty gold buttons, ranging from the seventh to the seventeenth transverse line, were measured by a student, after one hour's practice on other buttons. The weight of the thirty estimated by measurement was 3.465 milligr. and the weight on one of Lingke's blowpipe balances was 3.5 milligr. The result shows that, by averaging the readings even of five measurable buttons from as many assays of a rich enough ore, a very fair approximation can be made to the weight of the gold in the ore.

§ 164. The measurement can be also made with the microscope, as directed for silver, or by means of Rueger's instrument. For the latter Hünich (*loc. cit.*) has given the following formula for gold buttons. $D^2 \times 10.4326 =$ the weight of the button in milligrammes; D being its diameter in millimetres. If the measurement obtained by the microscope is in inches the student should remember, in using the above formula, that one inch = 25.399 millim.

IV. THE COPPER ASSAY.

This assay is simple, easy and as accurate as the furnace assay.

A. Ores and Products containing Sulphides, Arsenides and Selenides.

165. The substance is roasted, the oxide of copper reduced with addition of lead and the lead separated from the copper by refining with boric acid.

The Roasting.

Mix one ctr. of the fine powder thoroughly with 20 to 30 milligr. of very finely divided graphite (obtained by gentle scraping with a knife)

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in an agate mortar with the pestle. Minerals containing much antimony, bismuth, etc., like *bournonite*, fusing easily, are also mixed with 50 milligr. of powdered sesquioxide of iron (hematite will answer) to prevent caking. A clay capsule, § 139, is lightly coated with a wash of reddle and water and dried gently, after which the charge is spread thinly in it with the steel spatula, § 144, *a*. The capsule is then placed with the brass forceps on the platinum ring in a square coal, Fig. 62, prepared as follows:

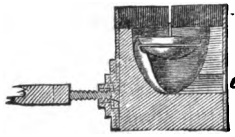

Fig. 62.

The coal being bored out until the top of the conical borer is even with the upper face of the coal, §§ 138, *d*, and 143, the latter is clamped in the holder, as directed in § 142. A hole is bored through the front of the coal at *a*, Fig. 37, with the spatula borer, § 10, *f*, and a slit cut above this, limited by the slit *b* in the coal holder, the door of which, *h*, is left open. The inner edges of the slit are also cut away somewhat, making it wider. The platinum ring and shield are then put into the coal, so that the ring is in the middle of the cavity, Fig. 39. A gentle O. F. is directed horizontally through the lowest part of the slit in the coal, until the capsule and coal are gradually heated to low redness, and this is continued until the odor of escaping fumes ceases, the heat being slightly increased during the operation, but not enough to fuse or cake the charge. This is transferred to the agate mortar with the spatula, § 144, *a*, ground fine and again roasted in the capsule, which is only recoated with reddle where this has necessarily been scraped off. The heat is raised somewhat higher than before and when the odors cease to be perceived on smelling carefully the still red-hot assay *and the graphite is almost entirely consumed*, the roasting is ended. A considerable residue of graphite would cause loss by sputtering during the subsequent reduction. Charcoal dust is not so good as graphite, but may be used, taking three volumes of coal dust for one of ore. After the coal has been consumed, as shown by stirring the charge with the steel spatula, previously warmed in the alcohol flame, to prevent the ore from adhering to it, the partially roasted ore is ground fine in the mortar and mixed with three volumes more of coal. It is then roasted at a somewhat higher temperature, until the odors cease and the coal is burned. Two roastings generally suffice, but if fumes still escape a third, or even a fourth addition of coal dust may be necessary, especially if much arsenide of nickel is present.

Roasted ores rich in oxide of copper are brownish-black, becoming

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lighter as they are poorer. Very thorough roasting is necessary to prevent possible loss by formation of sulphide of copper during the reduction, as this does not combine with the lead.

Reduction for Black Copper.

166. Weigh in the horn scale pans 100 to 150 milligr. of freshly ignited or *absolutely dry*, powdered soda, according to the amount of quartz or silicic acid in the ore, 50 milligr. of *dry* borax glass powder and 30 to 50 milligr. of lead. It is safe to add at least 30 milligr. of lead even with substances containing enough lead to yield a fusible alloy with the reduced copper, but carefully roasted ores containing much antimony, bismuth or tin, need no addition of lead. This charge and the roasted ore are well mixed in the agate mortar with the pestle and thence transferred first to the horn mixing capsule and from that to the soda paper, § 147, *a*, with the handle of the ivory spoon. The horn capsule is brushed out, the spoon handle wiped on the inside of the top of the paper cylinder and the charge fused in a coal crucible as in § 151, *b*. A very gentle R. F. is at first used, to prevent spirting, and when the fused metallic globules begin to collect the flame is directed only on the slag, turning the crucible holder constantly and using a spreading and moderately strong R. F., this process resembling closely the fusion of the silver assay. The charge must turn over at the proper time as described under that assay, the slag must preserve a spherical form and great care must be taken to bring the lead button into contact with every portion of the well fused slag and to continue the operation until all of the reduced copper globules are taken up by the lead. When this seems accomplished the assay is cooled, no O. F. being used at any stage.

The quite solid slag and button are removed from the crucible with the knife point, the coal brushed off and the slag carefully examined with the lens, above and below, for minute, detached globules of copper or other metallic particles. Should any be detected the whole is replaced in the crucible, a new one if necessary, and again reduced until all of the copper alloy is collected in one button (with the lead, if lead was used). The slag itself should be dark or gray, but if it shows a reddish color, due to suboxide of copper, it is reduced again with addition of 50 milligr. of dry soda, before removing the lead or other metallic button.

When ready the slag is separated from the black copper button by hammering between paper, or if the button contains any antimony, bismuth, tin, arsenic or sulphur, rendering it brittle, it is removed

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with the forceps when solid from the still soft slag, after which the adhering slag is carefully detached. The slag should be preserved and examined, since a second reduction of it will be necessary if any metallic particles are seen in it.

If the lead button appears dull gray or blackish it probably contains sulphur, due to imperfect roasting, or to sulphate of calcium, barium, or lead, either originally present in the ore or formed during the roasting. These cannot be freed from sulphur by roasting, and when they are present the reduction must be conducted slowly, so as to volatilize the sulphur as sulphide of lead. A small admixture of sulphide can thus usually be removed, adding a little more lead and continuing the fusion for some time. When there is much sulphide of copper and lead, forming a crust around the button, the latter is remelted and taken when solid from the still soft slag, after which it is fused in R. F. with twice its volume of lead and some borax glass, directing the flame afterward upon the glass, until the lead button appears lustrous.

The black copper button is refined as directed in § 168 or 169, according to its composition.

B. Ores and Products containing Oxide or Chloride of Copper, with or without Metallic Copper.

167. Such are *carbonates, silicates, slags, paints,* etc. Excepting the sulphates and poor slags, one centner of the substance is charged and fused as directed for the roasted ore, § 166, adding 20 milligr. of fine iron filings when phosphates are present, and omitting the lead, if enough is already present, unless the assay also contains nickel.

Sulphates are dissolved (2 ctrs.) by boiling with water in a porcelain vessel, adding a drop or two of nitric acid to convert iron salts into the ferric state (sesquioxide of iron) and an excess of solution of caustic potassa is added, the liquid almost boiled for a few moments, the precipitate well washed on a filter with hot water, removed from the filter, dried in a porcelain dish, the filter ash added, and the whole then treated like an oxide, as above.

Poor Slags need the addition of 50 to 80 milligr. of gold in one or two pieces, without any lead. The gold melts to one button, which must be very carefully brought into contact with all of the melted charge. The copper is all taken up by the gold and its amount determined by the gain in weight of the gold button. Should, however, any lead oxide be in the slag, this is also reduced and collected with the gold, from which it must be separated as described for cop-

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per and lead, § 168. Slags containing nickel oxide as well as copper, yield a grayish alloy of gold, nickel and copper, rather hard and brittle. After removing any lead if necessary, § 168, the gold button is weighed and fused on coal with borax, so that the blue inner flame touches the borax, whereby the nickel is oxidized and colors the borax brown. Although slowly separated, the nickel can thus be removed, if care is taken, without losing copper, the amount of which is determined by again weighing.

C. Alloys of Copper.

Black Copper from Assays.

168. The lead and copper alloy from the assay of true copper ores is to be treated as immediately follows (the alloy from lead ores containing less copper may need to be first concentrated, as in § 169, *a*): The button obtained by the reduction, §§ 166, 167, is placed on a coal capsule, § 138, *c*, in a shallow cavity about 4 millim. deep and 8 wide, made with the conical borer. Ordinary charcoal may be used, but is not so readily handled. Beside the button is placed fully half its weight of fused boric acid, in one or two pieces only (nearly the full weight of the button being taken if much lead is present) and the alloy is fused within the tip of the inner flame, p.30. As soon as it melts the inner flame is directed upon the melting boric acid, which is covered by it, leaving the alloy exposed. Care must be taken that the button does not run under the boric acid, from which it can only be made to emerge by properly inclining the coal and blowing more strongly. The alloy must be in contact with the coal on one side and the glass on the other, while too strong a heat must be avoided, as it may cause loss by spirting. Under these conditions the lead will oxidize and dissolve in the boric acid, while the copper will not, until the lead is all separated. When the melted alloy loses the dull red appearance of red-hot lead and begins to show the greenish color of melted copper (with which the student must become familiar) a more spreading flame is directed upon the glass, so that the remaining lead may be more slowly oxidized and the button may not spirt, which can happen if the cavity is too narrow. As soon as the metal shows the bluish-green color of pure melted copper the blast is stopped and the button lifted when solid from the still soft slag with the brass forceps. It must be copper-red and malleable enough to be hammered out quite thin (to a circle of two or three times the diameter of the button) without cracking. If it cracks badly a light gray spot can generally be detected in the crack with

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the lens, showing lead. In this case place the button beside the old boric acid, or some fresh, fuse it in R. F. and cautiously repeat the refining. This repetition is to be avoided if possible, since oxide of copper is now very apt to be taken up by the glass.

The boric acid glass after the refining should be yellowish and transparent, or possibly enamel-like in spots, but must show no sign of red, due to sub-oxide of copper. If so, the surest way to recover the copper from it is to crush the slag coarsely and treat it with a strong R. F. in a moderately deep cavity on coal, until all of the copper and some of the lead are reduced and gathered into one button, leaving the glass free from any red color when cold. If no lead seems to be reduced add a very little soda, so that a little lead may be reduced and a more fusible alloy formed. This lead is treated as before with boric acid and the copper button thus obtained is weighed with the first one.

The practiced operator may also recover the copper simply by treating the red glass at once, after removal of the refined copper button, with a good R. F., gathering up the little grains of reduced copper by then replacing the larger button in the boric acid, fusing it with a strong R. F. and causing it to float all around in the glass. They may also be recovered by powdering and washing the glass. If, however, the boric acid is rather highly saturated with lead oxide some lead will also be reduced and this must be separated from the copper by using fresh boric acid. Any silver in the ore will remain in the refined copper, from which it is separated by cupellation, § 159, *b*, after fusion with 15 parts of test lead.

Other Alloys Containing Copper.

169. *a. Copper and lead.* If much copper is present follow § 168. If very poor in copper one centner of the alloy is treated with its weight of boric acid, as in § 168, until the glass is so far saturated with oxide of lead that globules of reduced lead appear about its borders, when the lead is cooled and the pure copper obtained by refining with fresh boric acid. Should the original lead not be in one piece it must be melted to a button beside borax in R. F. before concentrating. The amount of lead is determined by loss. If silver is to be accurately determined some of the original alloy must be cupelled. See also *c*.

b. Copper and bismuth. The alloy from the assay of bismuthiferous copper minerals, to which no lead has been added, is fused alone on coal in O. F., until nearly all of the bismuth is expelled, after

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which a little lead is fused with it and the copper refined with boric acid.

c. Copper, lead, antimony, arsenic, iron, nickel, cobalt, zinc, etc. Black copper from smelting ores may contain all of the above impurities. One ctr. is fused on coal in R. F. with 30 milligr. lead, in one piece (or the granulated lead is used, if the alloy is very brittle, the whole charge being wrapped in soda paper) and one-half spoonful each of soda and borax glass, until the metals are well alloyed. The resulting button is refined as in § 168. Iron, cobalt and some nickel, with part of the lead and zinc, oxidize and dissolve in the boric acid, while some lead and zinc, with most of the antimony and arsenic volatilize; but if much nickel is present some of it may remain, covering the copper button with a thin film which impedes the refining. To remove this melt the button with about its weight of lead in one piece and again refine the copper. Should the boric acid, after the removal of the refined copper, show a red color, it is treated with R. F. and the reduced copper collected by grinding and washing the glass in the agate mortar, adding its weight to that of the copper button. Nickel and lead may also be reduced from a slag saturated with oxide of lead, and then the metal must be collected in one button and again refined for copper.

d. Copper, iron and zinc; some varieties of brass and black copper. One ctr. of the finely divided alloy is fused on coal in R. F. with ½ to 1 ctr. lead, according to the amount of iron and zinc present, and one-half spoonful each of soda and borax glass, until the metals are well alloyed to one button, which is then refined, observing the remarks in respect to nickel, *c*, and silver, *a*.

e. Copper with much nickel. See directions under nickel and cobalt, § 181 *et seq.*

f. Copper and antimony. The alloy obtained by assaying antimonial copper ores without adding lead is heated in a moderately deep cavity on coal in O. F. (gently to avoid spirting) occasionally directing the flame a little aside, to admit air to the alloy. The antimony volatilizes slowly and when the melted copper assumes the bluish-green color of pure copper it is cooled and hammered flat. If it is not pure the refining is repeated. The cavity must not be too deep or the alloy may spirt. All the silver in the ore will be in this button, see *a*.

g. Copper and tin. Bell metal, bronze, gun metal and the alloys obtained by smelting ores containing copper and tin. A flux, kept in a tight bottle, is made by fusing in a platinum crucible, or on coal, 100 parts by weight of soda, 50 of borax glass and 30 of finely

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powdered quartz. About 50 milligr. of the alloy, or all of it, if from the assay of an ore, is placed with about 60 milligr. of the above flux previously fused to a bead on coal, and the two are melted together with the R. F. The melted alloy begins to rotate and a broad, more oxidizing flame is then directed upon the glass alone, but so as to keep the air from it. This is done to oxidize and dissolve the tin and any iron present, leaving the copper behind. The button is kept in contact with the coal on one side and the flux on the other, until the flux is saturated with binoxide of tin, becoming enamel-like and showing minute globules of reduced tin. The button is then lifted from the slag, as soon as it is solid enough, with the forceps and fused beside fresh flux, until it begins to show the greenish hue of pure copper, when a moderate R. F., not too spreading, is directed upon the glass until the copper appears bluish-green and pure, § 168. If not pure and malleable it is remelted with one-half as much fresh flux. Should the flux show any red color, due to suboxide of copper, it is reduced for a short time and the resulting copper weighed with the main button. It will be free from tin unless the slag was highly saturated. There will always be a little copper oxidized, but not necessarily over 1.2 per cent of the copper present. Any considerable amount of sulphur causes much copper to be oxidized, hence tin pyrites must be carefully roasted.

V. THE LEAD ASSAY.

A. Sulphides of Lead.

First Method (Precipitation).

170. This simple method is used with all ores containing the lead as sulphide, with or without arsenic or antimony, and for lead matts, slags and accidental furnace products containing sulphide of lead. If the substance contains an appreciable amount of antimony it is gently roasted without any addition of charcoal (but if very fusible with 50 milligrm. powdered hematite) in a clay capsule, § 165, until the antimony fumes cease. This residue, or the raw material, in absence of antimony, is shaken down in a clay crucible, § 139, resting in the ring, § 141, by tapping it on the table, and on it is placed a bit of iron wire weighing from 25 to 50 milligr., according to the sulphur present. Over this is evenly spread 300 milligr. Plattner's flux, with a cover of three heaped ivory spoonfuls of dry salt. The ore and charge are only mixed when much of the charge (earthy matter, etc.) is to be converted into slag and the mixing is done before the salt

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cover is put on. Ores containing less than 10 per cent. of lead require one carefully weighed silver button of 50 milligr. or so, to collect the lead. The iron combines with the sulphur and so much of the arsenic as does not volatilize; the earthy matters and most of the sulphides dissolve and combine with the flux, the starch exerts a reducing action and the salt cover prevents the volatilization and favors the collection of the lead, freed or *precipitated* by the iron.

The uncovered crucible is placed on an iron wire ring in a square coal prepared just as in § 165, except that the coal is not cut out above the hole *a*, and the door *h* is closed. A coal cover bored through with the spatula borer, is placed on the square coal, Fig. 63, and care must be taken that the crucible is properly adjusted, so that not only the point, but the lower part also, is seen through the hole *a*, yet not so as to obstruct the passage entirely. A strong O. F. is then directed horizontally through the hole *a*, keeping the tip of the blue flame just without the coal and not heating the crucible too quickly. In a minute or two the charge begins to melt and make a bubbling noise, on the cessation of which a stronger blast is used for two or three minutes, taking care not to touch the crucible directly with the flame, lest it be eaten through by the alkaline flux. After the charge has come to complete and quiet fusion a powerful O. F. must, however, be directed for a minute or more upon the lowest part of the crucible, to melt all the lead to one button, or the assay will fail. The whole operation requires about six minutes and may be very conveniently watched if the long tube of the blowpipe is replaced by a rubber tube, the mouth-piece being held by the left hand against the lips, see § 147, *g*, 4.

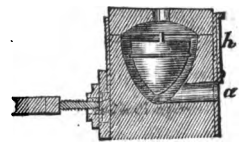

Fig. 68.

The blast is then stopped, the coal cover removed and the coal holder gently tapped to cause any floating lead globules to sink through the slag, which, with the salt cover, must be *perfectly fluid.* The crucible is now removed to the clay-crucible stand, and when quite cold is broken by striking it with the chisel edge of the hammer, held parallel to the ridge formed by the mould on the sides of the crucible. Care is sometimes needed to discover the lead button, which in a good assay will always lie at the bottom of one of the halves of the crucible and usually adhere to the remainder of the iron wire. The two are placed on the anvil, the iron, with possibly a crust of sulphide of iron, above, and are separated by careful hammering, after which the lead is cleaned by hammering it between

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filter paper; at first thoroughly moistened paper and then dry. Should lead adhere to the iron wire it is similarly cleaned and then carefully cut off.

The results correspond very closely with those of the furnace assay, both being too small, because the slag dissolves some sulphide of lead. The lead will only contain all the silver originally present in the case of galena free from other sulphides. Substances containing much sulphide of copper, or oxide of copper with comparatively little sulphur, will yield copper and lead, which must be separated. Antimonial ores must be roasted, as already stated, and then small quantities of copper are kept from going into the lead by mixing the roasted ore in the crucible with 100 milligr. of sulphur and heating the latter gently in the square coal furnace, until the blue flames cease.

Second Method (Roasting and Reduction).

171. This rather complicated method is recommended only for the simultaneous determination of lead and copper. The loss of lead, always from one to three per cent., may be greater if the fusion lasts too long. The ores and products thus assayed contain sulphides of lead and copper, with or without arsenic and antimony.

One centner of the finely divided substance is roasted with charcoal dust just as directed for copper ores, § 165, adding the hematite if necessary. The second roasting is made with two volumes of coal dust, any considerable amount of arsenic causing a strong odor as the coal burns away, in which case a third roasting with fresh coal dust may be necessary, especially in presence of arsenides of cobalt and nickel. When the ore yields, during the burning of the coal, at most only a faint odor of sulphurous oxide (or acid) the roasting is ended. The powder should then be loose, dull and quite free from shining, metallic particles. It is well mixed in the agate mortar with one centner of *dry* soda, and 30 to 40 or even 50 milligr. of borax glass, according to the amount of matter to be slagged off, the whole being then transferred to the horn mixing capsule and thence to a soda-paper cylinder, in which it is wrapped as for the silver assay, except that the corners of the roll are pressed down upon the sides of the paper cylinder. This is put into a well-dried lined crucible, § 148, *j*, over which the clay roasting capsule is inverted, after covering the charge with enough charcoal dust to fill both crucible and capsule. The crucible is then placed in a square coal, Fig. 64, just as in § 170, and over the coal is placed a perforated coal cover. The crucible is heated with O. F., as in the first method, a feeble flame issuing soon from

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the hole in the cover. Eight minutes heating suffices and then the wire ring and crucible are lifted out and set on the clay crucible stand. The charge should be melted to a ball, scarcely adhering to the coal lining, while no indications of yellow oxide of lead must be seen about the hole in the cover, indicating too strong a heat. The slag is separated from the lead, which is generally in several buttons, by hammering between paper on the anvil, picking out the larger buttons and then separating the smaller ones by carefully rubbing the slag in a porcelain dish with the agate pestle, repeatedly washing away the lighter slag and finally drying the residue of lead in the dish. The large buttons can be hammered and cleaned with water.

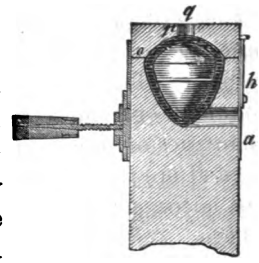

Fig. 64.

This lead should contain only copper, bismuth, antimony and silver. Copper is determined as in § 168 or 169, and the copper may be cupelled for silver, which will, however, be less than the true amount in the ore. To determine any bismuth, make a new assay and separate the lead as sulphate as in § 175. The lead, copper (and silver) being deducted give the weight of bismuth in the ore. However thorough the roasting, a little antimony will remain in the lead, but it need not become important and can only be determined by the wet way.

B. Lead Oxides, their Salts and Lead Chloride.

172. Here belong slags free from sulphides, cupel bottoms, skimmings of oxides, glass, etc. One centner of the substance is charged and fused just as in § 170, using only 25 to 30 milligr. of iron wire, adding 25 to 30 milligr. of starch powder, mixing the whole and tapping it gently to settle the charge before putting on the salt cover. Silver is added if need be, while a little copper is kept from entering the lead as in § 170.

C. Selenides and Tellurides.

173. These may contain other metals. They are assayed as above, omitting the starch. Any silver or gold will go into the lead and may be obtained by cupellation. If copper is present part of it will pass into the lead, from which it must be separated, §§ 168, 169, after the lead is weighed.

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VI. THE BISMUTH ASSAY.

A. Ores and Products containing Free Bismuth, perhaps mixed with earthy gangue, or Arsenides of Cobalt, Nickel or Iron, or containing Selenides and Tellurides of Bismuth.

174. Cobalt and nickel ores and speisses containing bismuth are included here. If the substance yields arsenic in the closed tube, one ctr. in fine powder is heated in a clay crucible, resting on an iron wire ring and covered with a clay capsule, in the square coal, just as in § 171, keeping the crucible at a low red heat until the arsenic fumes cease (the arsenic fumes should not escape into the laboratory), and lifting it out to cool *with the cover on*. With the cold residue, or the original powder, should it not require this heating, are mixed a bit of iron wire weighing 25 to 30 milligr., 50 to 200 milligr. of pure silver in one or more bits, accurately weighed, and 300 milligr. Plattner's flux, the whole being settled by tapping the crucible on the table. If the ore has been caked by the heating, the charge is necessarily only placed upon it. After adding a cover of three spoonfuls of salt the assay is fused as in § 171, for five minutes, or six at the most, and the metallic button is extracted as in § 170. If much arsenic was present the button may be quite round and will really consist of two parts, a white alloy of bismuth and silver, with an adherent gray, metallic speiss, or arsenide, of cobalt, nickel and iron. The alloy, which is less brittle than bismuth alone, is carefully freed from the arsenides by hammering between paper and then fusing it cautiously beside a little borax glass with a feeble R. F. (to avoid loss of bismuth) in a moderately deep cavity on coal, but only until it becomes bright, after which the glass is removed by hammering. Should the substance contain silver the alloy must be cupelled with one ctr. of lead and the weight of the remaining silver deducted to determine the bismuth. Where less arsenic is present some metallic iron, perhaps coated with sulphide of iron, may adhere to the alloy, the shape of the two being then not so spherical, and this iron is to be separated between paper as before.

B. Ores and Products containing Sulphide of Bismuth, alone or with other Sulphides.

175. When sulphide of antimony is present one ctr. of the powdered substance is roasted as in § 165, and, in presence of copper, again sulphurized, § 170. This prepared powder, or otherwise one ctr. of the raw substance in powder, is charged and fused as in § 174.

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The alloy of silver and bismuth, melted to a button, is separated from the residue of iron and cleansed by fusing with borax glass, § 174. Antimony being chiefly expelled in roasting and a little copper going into the slag as sulphide, the alloy will contain little except bismuth, silver and lead, with some copper, if much was originally present. To estimate these fuse the alloy in a platinum crucible over the spirit lamp, with 12 to 15 parts of potassium bisulphate, adding more flux by degress, until the metal has quite dissolved. Then cover the *cool* crucible with water in a porcelain dish, heating it until the fused mass is entirely disintegrated, allow the insoluble powder to settle, decant the *quite clear* solution upon a small weighed filter and wash the residue again by decantation, keeping as much as possible of the insoluble sulphates of lead and bismuth in the dish. They are covered in the dish with water containing a little concentrated sulphuric acid and a drop or two of nitric acid, boiled a moment and presently decanted upon the same filter (see note) and washed first with water containing a little sulphuric acid, afterward with alcohol until all acid is removed, throwing away the alcohol washings. Insoluble sulphate of lead remains upon the filter, containing 68.3 per cent of pure lead. If any notable amount of copper colors the filtrate from the sulphate of lead bluish, heat it, add carbonate of ammonia to alkaline reaction, collect the precipitate of carbonate of bismuth on a small filter, wash it well with hot water, dry it and reduce it as in § 176, without adding iron.

The results are reasonably accurate even in presence of copper, but less so when antimony is present, because this remains with the sulphate of lead. Should the substance itself contain silver this must be determined by a separate assay and deducted.

Note. A *weighed filter* is prepared by drying a filter at 100° C., until it ceases to lose weight, weighing it always inside of a little glass tube, with a cork stopper, within which it is also to be cooled. The precipitate and filter are similarly dried and weighed, the increase of weight representing the weight of the precipitate. When acid liquids are to be poured through the filter this should first be prepared by pouring some dilute hydrochloric acid over it on the funnel and thoroughly washing and drying it before taking its weight.

C. Oxide of Bismuth.

176. This method is used for oxides and their salts, and chloride of bismuth, with perhaps oxidized compounds of other metals. Substances free from oxides of copper, nickel or cobalt, are treated as in § 174 (but with only 80 to 100 milligr. silver), otherwise they are first sulphurized, § 170, and then treated as in § 174; or they may be

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treated as described under the nickel and cobalt assay, § 181, *et seq.*, to determine cobalt, nickel and copper also.

In the treatment of some tin ores a basic chloride of bismuth is obtained which may contain a little binoxide of tin, and this yields an alloy of bismuth and tin, the latter readily recognized by the crust which forms on the surface of the silver and bismuth button during the cleansing fusion with borax glass. The weighed alloy must then be hammered thin and treated with nitric acid containing one-fourth its volume of water, the solution diluted and warmed, the insoluble residue of binoxide of tin collected on a small filter, washed, dried, ignited in a very thin porcelain capsule, or in a platinum spoon, with proper precautions to avoid injuring the platinum, and weighed. It contains 78.6 per cent. of tin, but not all of the tin originally present in the assay.

VII. THE TIN ASSAY.

A. Sulphides, or Oxides containing Arsenides or Sulphides of other Metals.

177. *Stannite* (*tin pyrites,*) *tin slimes*, from the dressing of oxidized ores containing sulphides or arsenides, and *mosaic gold.*

One centner of the powdered substance is well roasted with charcoal dust, exactly as directed in § 165, two or three roastings being necessary if much arsenic or sulphur is present. Substances belonging in this class, after roasting, generally contain a considerable amount of oxides of iron, bismuth, copper, etc., which must be removed or they will pass into the tin during the reduction. For this purpose the roasted charge is next well covered with hydrochloric acid in a porcelain dish and gently boiled for four or five minutes. The above oxides, with any zinc or antimony, are dissolved, and after standing a short time the solution is diluted with water (which may consist of the rinsings of the glass cover), the insoluble residue filtered out on a small filter, washed, dried, removed from the filter, gently ignited in a porcelain crucible and the filter ash added to it. If there is very little residue it may be ignited with the filter. In presence of bismuth the solution, after settling quite clear, should not be diluted, but is at once drawn off from the residue with a pipette, as completely as possible, and then water is used to wash the residue three or four times, being poured down the side of the dish so as not to agitate the residue, after which it is drawn off, keeping the end of the pipette far enough below the surface to avoid drawing off any of the fine particles floating on the surface of the water, which must be gently warmed. Dry the residue and reduce it, or the resi-

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due with the filter ash above, as will be presently directed. Slimes already once roasted on the large scale and known to contain very little sulphur or arsenic, need not be roasted, but may be at once treated with acid as above, using however one part of strong nitric acid and two of hydrochloric.

The prepared material is reduced in a lined clay crucible as in § 171, being wrapped in a soda-paper cylinder with a charge of 100 milligr. of *dry* soda and 30 of borax glass, the whole having first been mixed in the agate mortar. At least ten minutes will be required for the fusion, which must be conducted at a high temperature, and automatic apparatus will be found very useful, in connection with Hirschwald's or Fletcher's furnace. A perfectly good assay can, however, be made with the ordinary apparatus. After the assay has cooled a ball will be found at the bottom of the crucible, consisting mainly of slag, containing the tin, usually in a number of little buttons, which are freed from the slag by crushing the ball between paper, or in the steel mortar, as fine as the disseminated tin buttons will admit, pouring the powder into a large enough porcelain dish and warming it with water. The water is poured off when the slag has become well disintegrated and mostly dissolved, and the moist residue is rubbed with the agate pestle and washed carefully, to remove the coal, etc., from the tin. Quartz may render the slag insoluble in water and in this case it is first warmed with acetic acid.

The separated tin is finally dried in the dish, after which its purity can be determined by its color and malleability. If the soluble oxides were not properly removed by the acids before the reduction the tin may be brittle, owing to the presence of antimony or copper, or it may, although remaining malleable, follow the magnet under water, owing to reduced iron. In either case the result will be too high. Small quantities of tungsten or titanium will have been slagged off during the reduction, but when much tungsten is present the tin will unavoidably contain some of it, for these two substances are not removed by the treatment with hydrochloric acid.

The automatic gas blowpipes are especially useful in the tin assay, where they generally secure the fusion of the tin into one button.

B. Oxides free from much Iron Oxides and from Arsenides or Sulphides.

178. *Cassiterite, artificial binoxide of tin, and enamel.*

The first two, unless accidentally containing oxides of copper, iron or antimony, are not treated with hydrochloric acid, but one ctr. of the dry, finely powdered substance is reduced at once just as directed

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for the prepared residue in § 177. *Enamel* contains silicic acid and requires a charge of 150 milligr. of soda with 30 of borax glass. The resulting tin will also contain any lead which may have been in the enamel and the tin must then be heated with slightly dilute nitric acid, which finally dissolves the lead and leaves oxide of tin behind. This oxide is collected on a filter, well washed, dried, separated from the filter if possible, the filter ash added to the oxide and the whole strongly ignited in a porcelain or platinum crucible, after which it is weighed as binoxide of tin, containing 78.6 per cent. of tin. If it cannot be separated from the filter the whole is ignited in a porcelain crucible.

C. Oxides containing Silicates of Iron and the Earths.

179. *Slags* of various sorts are included here. They may contain not only combined tin oxide, but also unaltered tin ore and free tin. Slags containing free tin are powdered and from two to five grammes are weighed out, the larger globules of tin picked out from the powder, and the residue ground and washed in the agate mortar, until all the true slag has been washed into a beaker. The residual tin in the mortar is dried with the well washed larger globules in a porcelain dish, while the washed (elutriated) slag is collected on a filter, dried and treated as below. Slags containing no free tin should also be rubbed with water in the mortar and elutriated, until all is reduced to the finest powder, since in this condition alone can they be properly treated.

Fifteen to eighteen ctrs. of potassium bisulphate, roughly weighed, is then brought to quiet fusion, in a covered platinum crucible, at a moderate heat over the spirit lamp, and one ctr. of the elutriated and perfectly dried slag is thrown into it by little and little, gradually increasing the heat, until the red-hot, fluid salt is free from undecomposed slag and ceases to bubble. The cold crucible is then covered with water in a porcelain dish, a few drops of hydrochloric acid are added and the whole heated until the potassium salt is dissolved, together with sulphates of iron, copper, etc., while the insoluble residue of oxide of tin, with silicic acid and tungstic acid, if any of the latter was present in the slag, is collected on a filter, washed, dried, separated from the filter if possible, ignited in a porcelain crucible and reduced, together with the filter ash, as directed for enamel, § 178.

D. Alloys.

180. Alloys of tin with copper, if free from lead, bismuth, or anti-

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mony, can be assayed by separating the tin from the copper according to § 169, *g*, the slag containing the tin oxide being carefully preserved, finely powdered, mixed with fifty milligrammes of soda, wrapped in soda-paper and further reduced in a coal lined crucible, as in the ordinary tin assay. The tin will usually contain traces of copper.

Alloys containing lead, bismuth or antimony must be treated by the wet way.

VIII. THE COBALT AND NICKEL ASSAY.

Preliminary Remarks.

181. These two metals are rarely found apart from each other either in ores or metallurgical products and they are also frequently accompanied by copper, lead or bismuth, which can be determined at the same time. The cobalt and nickel are separated as arsenides from the substances assayed, the metals themselves being too infusible. The arsenides are then subjected to an oxidizing fusion, which effects the successive separation of iron, cobalt and nickel from one another, the iron and any excess of arsenic being first removed, while the cobalt and nickel remain as arsenides of definite composition.

If a fragment of such a compound, containing little iron and weighing from 60 to 80 milligr. is fused beside borax on coal in a shallow cavity within the tip of the inner flame, until the metal button assumes a rotary motion, and if then the O. F., not too hot, is directed upon the fused borax bead, the iron arsenide oxidizes and covers the surface of the button, free from the glass, with a thin film of basic iron arsenate, which is brought by the rotary motion of the button into contact with the borax and at once dissolved by it, while a part of the arsenic which was combined with the iron and the other metals escapes in odorous fumes. If the operation is stopped when the not too highly heated button shows a bright surface, which no longer becomes covered with an oxidized film, although still more arsenic fumes escape, and the solidified button is removed with the forceps from the still fluid slag, the latter will be black and a bit of it fused on platinum wire in O. F. will show only iron reactions; the cold button will, however, have become covered with an almost black film of oxides. If much iron arsenide was present one fusion will not separate it, but it must be treated with fresh borax, until it is bright and begins to give off arsenic fumes; after each fusion the solidified button is to be removed with the forceps from the soft borax, when this is saturated, and thrown into a little water, after which the cracked glass is rubbed from the button with the fingers or between paper.

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If the clean button is now fused with a gentle R. F. in a shallow cavity on coal, its surface remaining bright, until no more arsenic fumes are visible, there will remain a definite compound* containing Ni_2As (with 60.73 per cent. of nickel) and Co_2As (with 61.14 cobalt), the proportion of nickel to cobalt varying according to the nature of the substance treated. When this compound is again oxidized beside borax, as just directed for slagging off the iron, the cobalt arsenide oxidizes and the cobalt dissolves as arsenate in the borax, while very little or no arsenic odor will be perceptible, if the excess of arsenic had been properly expelled. The surface of the button will, however, remain bright if the same temperature is maintained as during the oxidizing of the iron, and the borax glass will show a smalt-blue color when the blast is stopped and a little of the glass drawn out into a thread. Should the heat be too feeble a very transient film may occasionally pass over the still melted button and the solid button, if still containing cobalt (which, moreover, separates much more slowly than the iron) will be covered with a black film, as after the separation of the iron. On again fusing the button at the right temperature beside fresh borax glass its surface will remain bright so long as it contains cobalt arsenide, but when this is all removed and nickel arsenide begins to oxidize a film of nickel arsenate will form and slowly move over the surface of the button, until it is finally dissolved by the borax. This appearance will continue so long as the oxidizing process is kept up and the glass can still dissolve the nickel arsenate. To observe it, however, one must know what temperature to maintain, since too high a heat, more than was used in oxidizing the cobalt arsenide, may easily cause the pure nickel arsenide to show a bright surface. If the blast is stopped as soon as the nickel arsenate film is observed and a little of the glass at once flattened between the forceps and slowly drawn partly away from the rest of the glass, it will usually, in bright day-light, appear rather violet than blue, provided the glass is not too highly saturated with cobalt. Had all of the cobalt been previously removed the glass would only have appeared slightly brown (nickel). In addition to this violet color of the glass may be noticed an apple-green film of nickel arsenate on the button, showing likewise that the latter is now only nickel semi-arsenide. The cobalt semi-arsenide is slagged off without leaving any of its arsenic combined with the nickel semi-arsenide and the two metals can therefore be quantitatively determined. The loss of nickel need be but trifling, if proper care is exercised; even when much nickel is present, the whole surface of

* For the sake of convenience called semi-arsenides.

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the button must be covered with the apple-green nickel arsenate film and the borax glass also show a little nickel reaction, before the loss of nickel reaches 0.5 milligr.

When copper is also present the nickel is not so easily estimated. Provision must first be made for converting the copper also into arsenide and this finally remains as Cu_3As (71.72 of copper) with the nickel and cobalt semi-arsenides. This proportion of arsenic to copper only remains constant, during the separation of the other two metals by borax, so long as there remains at least as much nickel arsenide as there is copper arsenide, but otherwise some of the arsenic previously combined with the copper volatilizes. A good result can, however, be obtained, after separation of the cobalt arsenide by fusing the copper and nickel arsenides (provided there is less copper than nickel) with a carefully weighed gold button of 60 to 100 milligr., more or less according to the copper present, and slagging off all the nickel arsenide, leaving the fused gold and copper alloy with a bright surface and the bluish-green color, peculiar to these metals when melted. The button must show a bright surface also when cold, and all arsenic will have been removed from it. Previously fused S. Ph. must be substituted for the borax, which dissolves the nickel arsenate too slowly, and the operation is conducted just as already directed for the separation of cobalt arsenide from the nickel arsenide. A gentle O. F. is used and when the S. Ph. is saturated the button is removed, cleansed after dropping it into water as before, and treated with fresh S. Ph. When the melted button ceases to show the oxide film and begins to assume the bluish-green color of pure gold or copper, borax is employed, to remove the last traces of nickel arsenide and arsenic with less danger of oxidizing the copper. The remaining button, if bright and sufficiently malleable, is weighed and its increase in weight gives the amount of copper present. The weight of Cu_3As corresponding to so much copper is calculated and this deducted from the weight of the copper arsenide and nickel arsenide already found, the remainder being the weight of Ni_2As present. The determination is not absolutely exact, for some copper is oxidized, but it is close enough to render the assay often desirable ; compare § 183.

A. Ores and Products containing Arsenide of Cobalt and Nickel, with perhaps a very little Sulphur, but free from Copper; Bismuth may be present and can be estimated at the same time.

182. Here are included native arsenides and also the speisses produced from nickel and cobalt ores proper.

The preliminary fusion of the assay for the arsenides can be made

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in two different ways ; either in a clay crucible, whereby all mechanical loss is avoided and there is less liability to oxidation of cobalt in absence of iron arsenide, or in a cavity on coal. The first method if undertaken with ores containing much cobalt arsenide may necessitate the addition of iron arsenide to render the arsenides more fusible. It will be presently described.

When the second method is followed one ctr. of the powdered substance is mixed in the agate mortar with 50 milligr. of soda and 15 of borax glass, packed in soda-paper and fused in a coal crucible as usual, with a pure, moderately strong R. F. until the fluxes form a perfect slag and all the arsenides are collected in one button. The button is lifted from the slag and thrown into water to cleanse it, after which it is fused in a shallow cavity on coal with a moderate R. F. Should its surface remain bright while it is melted it contains no iron and it will only be necessary to keep it thus fused until no more arsenic fumes escape, after which it is allowed to cool and is weighed, as a mixture of Ni_2As and Co_2As, if both are present. During the fusion the R. F. must only be strong enough to keep the metal bright, otherwise it may spirt ; any trifling amount of bismuth will volatilize with the excess of arsenic.

If the button does not remain bright while fused, but is quickly covered with a film of oxide the iron arsenide causing this is slagged off as in § 181. A little loss of cobalt is here unavoidable, for if any iron is left the excess of arsenic cannot be expelled, as it should be before weighing the Ni_2As and Co_2As ; see § 181. The Co_2As is next slagged off and the resulting Ni_2As weighed. When the necessary experience is wanting the student can control the operation by fusing the weighed Ni_2As button beside fresh borax for a short time and if the glass should show still a pure blue color, instead of the brown of nickel or the violet of nickel and cobalt, the button should again be weighed, when all of the cobalt has been removed. It may happen that the borax will become saturated with cobalt arsenate before all of the cobalt arsenide has been removed from the button, in which case the latter is freed from slag as usual and treated with fresh borax, as often as necessary, but using diminishing quantities of borax every time.

Any silver present will remain as sulphide with the Ni_2As, so that this, after weighing, should be fused with enough test-lead beside borax on coal in R. F., the lead cupelled and the weight of the silver, calculated as Ag_2S (100 Ag yield 114.8 Ag_2S) deducted from the weight of the Ni_2As.

When the first fusion is made in an unlined clay crucible the pow-

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dered ore is placed in the crucible, freed, if necessary, from any great excess of arsenic as in § 174, covered with 300 milligr. of Plattner's flux, the whole covered with salt and fused as in § 170.* After the fusion a single button of the arsenides must be visible at the bottom of the perfectly fluid, clear slag. This button, freed from slag after breaking open the crucible, is treated just like the one obtained by the original fusion in the first method.

When bismuth is to be determined simultaneously the powdered substance is treated exactly as described in § 174, but the brittle speiss of arsenides adhering to the silver and bismuth alloy is very carefully separated from the latter by hammering between paper or in the steel mortar, after which it is perhaps broken a little finer and then fused in a coal crucible with some soda and borax glass in R. F., and the resulting button of arsenides of nickel, cobalt, iron (and copper) treated as already described.

B. Assay of Sulphides, with or without Arsenides; Lead, Bismuth and Copper may also be estimated if present.

183. In addition to the ores naturally belonging here this class includes lead speisses, lead matts containing nickel and cobalt, and poor matts consisting mainly of iron sulphide. The presence of more than a very little sulphur necessitates a preliminary roasting of the assay. This is performed in a clay capsule as in § 165, at first with charcoal dust, but after the hot powder ceases to yield any odor it is rubbed in a mortar with fifty milligrammes of ammonium carbonate and again roasted. The assay is then powdered, mixed with one ctr. of finely powdered metallic arsenic, transferred to a clay crucible, mounted on an iron wire ring in the square coal, § 170, and covered with a clay capsule. The square coal cover, with a hole bored in it, is placed over the coal and the crucible finally brought to a low red heat. When arsenic fumes cease to escape the still covered crucible is lifted from the coal and allowed to cool. The arsenic fumes evolved in this operation of *arsenicizing* may prove injurious and when there is reason to avoid their escaping into the surrounding air the mixture of roasted ore and arsenic is transferred from the mixing capsule to a well dried glass tube, previously sealed at one end, and slowly heated to redness in the alcohol flame, so long as any arsenic condenses in the cool part of the tube. When cold the tube is cut off just below the sublimate with the file and the residue of arsenicized ore transferred to the clay crucible.

*If cobalt arsenide predominates 15 to 20 milligr. of iron arsenide, see footnote, p. 228, must be added before the Plattner's flux.

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Upon the substance thus prepared, by either method, and placed in the crucible, is put 300 milligrammes of Plattner's flux, which is pressed down with the spoon, covered with salt as usual and fused as directed in § 170, with a high temperature at the last, to fuse the arsenides into a single button. If lead or bismuth is present in any quantity and is to be estimated also, about 20 milligr. of iron wire and 50 to 100 milligr. of silver, the latter carefully weighed, are added, before the Plattner's flux and the fusion conducted according to § 170, the arsenides being separated from the alloy of silver with lead or bismuth between paper or in the steel mortar.

The alloy is further treated, if necessary, according to § 174, while the fragments of arsenides, with the slag last detached from the silver alloy, are fused with soda on coal in R. F. and the resulting button of arsenides treated according to §§ 181, 182. Should no bismuth or lead be present and no iron therefore required to separate them, it may happen, with substances containing chiefly cobalt, that the resulting arsenides will not fuse readily in the crucible. To such, therefore, 10 to 20 milligr. of iron, according as they are richer or poorer in cobalt, should be added before they are arsenicized, while if they are very poor both in nickel and cobalt, a collecting medium may be necessary. In this case either 15 to 20 milligr. of iron arsenide* should be added before the fusion in the clay crucible, or else 10 to 15 milligr. of iron filings can be added before the arsenicizing. Any such addition of iron is, however, unnecessary in case the roasted substance contains much iron oxides.

The button of nickel, cobalt and iron arsenides finally obtained from any of these operations is to be treated as in §§ 181, 182. Any copper present remains with the Ni_2As and is to be separated as in § 181. (See note at the end of this paragraph.) If considerable copper is present it can only be accurately determined by the wet way. The button of Ni_2As and Cu_3As, after removal of the Co_2As, is treated with a mixture of about three parts of dilute sulphuric acid and one of nitric acid, the copper and arsenic precipitated as sulphides from the hot diluted solution with hydrosulphuric acid, the sulphides filtered out, washed with water containing hydrosulphuric acid, the wash-water and filtrate boiled together to expel all hydrosulphuric acid, any arsenic sulphide which separates again filtered out and the nickel precipitated from the filtrate by treating with a not too concentrated solution of potassa, the precipitate collected on a little filter, washed, dried, ignited, and, together with the filter ash, mixed with 50 milligr. of soda and 25 of borax glass, an exactly

* Made by heating iron filings and metallic arsenic in a closed tube or crucible.

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weighed, small gold button added, the whole wrapped in a soda-paper cylinder and reduced as usual on coal. The gain in weight of the gold button represents the metallic nickel present. To determine the copper the precipitate containing it is dried, separated from the filter and heated, at last strongly, in a deep porcelain capsule until all the arsensic sulphide has been expelled. The filter ash is then added and the heating continued until the copper sulphide is mainly converted into sulphate. When no more odor of sulphurous oxide is perceptible six to eight times as much potassium bisulphate is added and the whole gradually brought to a low red heat and maintained in fusion until all of the copper is dissolved. The capsule may well be covered with platinum foil during this fusion. The cold mass is covered with enough water, boiled, filtered if necessary, and the copper precipitated as oxide and finally reduced and collected in a weighed gold button, as just directed for the nickel.

Note.—Any notable amount of silver that may be present in substances of this class should be deducted, either as silver sulphide from the Ni_2As, § 182, or as metallic silver from the bismuth, if that metal had been quantitatively determined. If the wet method, just described, has been followed, the silver can be precipitated from the acid solution by means of hydrochloric acid, before precipitating the copper and arsenic sulphides.

C. Oxides of Cobalt and Nickel with various Acids, or with other Metallic Oxides.

184. The general charge for the fusion of these consists of one ctr. of the fine powder in a clay crucible, to which 300 milligr. Plattner's flux is added, being pressed down on the charge and covered with salt as usual. Substances poor in cobalt or nickel, or containing more cobalt than nickel, will require the addition of arsenide of iron in some way as follows:

Annabergite (*nickel ochre*) may be at once charged as above. *Erythrite* and *cobalt bloom* will require the addition of 15 to 20 milligr. of iron arsenide, prepared by arsenicizing iron filings with arsenic, as directed for arsenicizing roasted sulphides, § 183. *Roasted cobalt ores* and *speisses*, *lavendulan*, *earthly cobalt* and *bieberite* (which must first be well roasted in a clay capsule with charcoal powder) are mixed with 15 to 20 milligr. of iron filings and then arsenicized, § 183, after which they are charged as above. Other minerals and ores, *emerald nickel*, *genthite*, *pimelite*, etc., belonging here, as well as *roasted nickel ores* and *speisses*, are arsenicized at once and charged as above. In case lead and bismuth are present in any quantity iron and silver,

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§ 183, must also be added before covering the assay with Plattner's flux.

The fusion is made as already described, § 183, from which, as well as §§ 181, 182, the rest of the operation can be learned.

D. Oxides of Cobalt and Nickel, with comparatively little of other metallic Oxides or Earths.

185. These are first ignited to expel moisture, after which 50 milligr. of the finely powdered oxide is to be arsenicized as usual, but with addition of 15 milligr of iron filings when cobalt oxide prevails, and fused with the same charge as directed in § 183, the subsequent treatment of the resulting arsenides being in all respects like that previously directed.

The arsenicizing may also be performed by wrapping the charge of oxides with 100 milligr. of powdered arsenic in a soda-paper cylinder and slowly heating this to redness in a glass tube over the spirit lamp, after introducing a roll of filter paper, Fig. 65, to absorb the moisture evolved from the charring paper. The tube is cut off below the sublimed arsenic, the charred soda-paper cylinder transferred, with the aid of a wire if necessary, to a cavity of proper depth in coal, covered with 50 milligr. soda and 20 borax glass and fused in a moderate R. F., directed at first on the flux and afterwards more strongly urged between the coal and charge, so as to fuse the arsenides from the bottom upward, while the charred paper is burned away. The operation is best ended by fusing the assay again in R. F. with addition of half a spoonful of potassium oxalate, as the slag will then be absorbed and leave the button free. Any small globules of arsenides detached from the larger button should then be placed by it and the whole melted together in the R. F. The resulting arsenides are treated as in §§ 181, 182.

Fig. 65.

E. Alloys, Arsenides and Sulphides, with more Copper than Nickel, or Alloys with Antimony.

186. *Breithauptile, black copper, copper matts* and *slags, German silver* and similar alloys.

Alloys of nickel and cobalt with antimony are decomposed with hot nitric acid, the insoluble antimonic acid filtered out, the hydrated

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oxides of nickel and cobalt precipitated from the filtrate by boiling with excess of solution of potassa, collected on a filter, washed, dried, ignited and treated as directed in § 185.

Substances containing more copper than nickel are decomposed with nitric acid, hydrochloric acid being added if necessary to effect the decomposition, the solution evaporated nearly to dryness with addition of hydrochloric acid to expel the excess of nitric acid, the solution diluted with water and treated with hydrosulphuric acid, filtered, the residue in the filter washed with water containing a little hydrosulphuric acid water, the filtrate and wash water boiled with a few drops of nitric acid, then boiled with excess of solution of potassa and the precipitate collected on a filter, washed, dried, ignited and treated as for oxides in § 185.

In the dried precipitate produced by the hydrosulphuric acid will be found the copper as sulphide, with sulphides of any lead, bismuth, arsenic or antimony present. The lead, bismuth and copper can be determined as directed in the various assays for these metals.

IX. THE MERCURY ASSAY.

187. *a. Domeyko's Method for Sulphides.* About 0.5 grm. of the ore is mixed with 0.75 to 0.8 grm. of finely powdered litharge and introduced into the lower end *a* of a glass tube, Fig. 66, which is from 16 to 18 centimeters long and 6 to 7 millim. wide, the lower, closed end *a* being about 3 centim. long. The lower end of the tube is gradually heated over a good alcohol flame, until the whole mixture fuses and the glass softens. Any water present condenses at about *b*, while the mercury forms a sublimate near the bend, and can be driven a little further off by lowering the tube in the flame. After the tube is cold it is cut with a file close below the mercury which is brushed into a horn capsule and weighed.

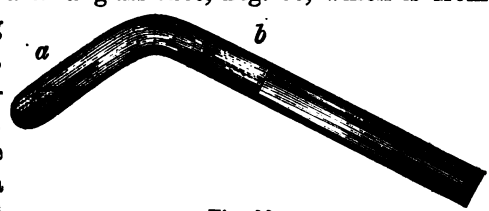

Fig. 66.

b. Küstel's Method. Of the poorer ores about 100 milligr., of the richer only 50 milligr., being introduced into a closed glass tube *a*, Fig. 67, similar to those used for qualitative tests, the ore is covered with three or four volumes of perfectly dry soda, *b*, and about 300 to 400 milligr. of very thin

Fig. 67.

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sheet gold, rolled into a spiral shape, placed next to the soda. The tube is held horizontal and the closed end heated at first in the alcohol flame, afterward with the blowpipe. The mercury is taken up by the gold and any particles which may have condensed on the glass are brought into contact and amalgamated with the gold by moving the latter, which is then withdrawn and weighed in a horn capsule. Any water that may have condensed on it is first driven off by heating the gold in a short wide glass tube or other vessel held in boiling water.

Küstel's method is adapted to chlorides as well as sulphides, Domeyko's is not, but Domeyko's answers best in presence of small quantities of oxidizable volatile substances, like arsenic and bitumen. When applying Domeyko's method to chlorides about five volumes of soda should be also used, since the mercury chloride is not decomposed by litharge alone. Neither method answers with ores containing *idrialite*, since it yields other volatile products which render the mercury impure.

c. Amalgams. If sufficiently free from sulphur, etc., these are simply heated, very gradually in a glass tube as above, to get the mercury as a sublimate; otherwise they will require the addition of more or less soda.

X. ANALYSIS OF IRON ORES (FOR IRON ONLY).

188. *a.* Only a rapid method for determining the iron with reasonable accuracy will here be given. A qualitative blowpipe or other test may be made for the silica, earths, metallic oxides, sulphuric acid, sulphur, phosphoric and arsenic acids, which often are found in iron ores. If any of the last four are found in notable quantity the ore may at once be regarded as likely to yield an inferior iron for general uses.

b. Preparation of the Ore.

The ore, broken small enough on the anvil, is powdered rather fine in an iron mortar, by striking it with the pestle, avoiding any unnecessary rubbing, which would abrade the mortar too much. Finally it is mixed, sample spoonfuls taken here and there and the sample ground very fine in the agate mortar and dried in a porcelain dish over a water bath.

c. Assay for Iron.

One ctr. of the prepared ore is digested in a porcelain dish with sufficient hydrochloric acid to dissolve all soluble matter, the dish being heated on a water bath or over the alcohol flame. If the acid causes much effervescence it must be gradually and carefully added

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and the dish covered with a watch glass. When all is dissolved that will dissolve (usually at least a little insoluble residue remains) the solution is boiled for a short time with addition of a few drops of nitric acid, then diluted, filtered, if any insoluble residue remains, and the latter well washed with hot water. If the residue is not pure white it should be dried on the filter, mixed with three volumes of soda and one of borax glass, the whole wrapped up in the filter and fused in a coal crucible in O. F. to a clear bead. This is pulverized and digested with dilute hydrochloric acid in a porcelain dish, after which a few drops of nitric acid are added and the whole evaporated to dryness. The residue is warmed with a few drops of hydrochloric acid for some minutes, then with water, filtered and the filtrate added to the original solution of the ore, to which a drop or two of dilute sulphuric acid is also added, which will produce a turbidity if barium salts are present. In this case the solution is heated and sulphuric acid added as long as any barium sulphate is thrown down, after which the solution is again filtered if necessary, ammonia added in slight excess, the whole boiled for a short time and the precipitate of iron and aluminium hydrates filtered out, washed with hot water, and the filter spread out on another filter. The precipitate is carefully scraped off with the steel spatula and put into a porcelain dish, after which the filter is laid in another dish, moistened and gently warmed with just enough hydrochloric acid to dissolve the residue of the precipitate and the solution transferred to the dish containing the main part of the precipitate, the filter paper being rinsed once or twice with a very little hot water, which is also poured finally into the first dish. The contents of the latter are now well covered with a concentrated solution of potassa which is then brought to gentle boiling for a short time, after which it is diluted and filtered off from the insoluble hydrated sesquioxide of iron (ferric hydrate). The potassa is used to dissolve out all aluminic hydrate as well as traces of silica. The ferric hydrate is well washed with hot water, dried on the filter, transferred as completely as possible from the latter to a weighed platinum or porcelain capsule, the filter burned to ash in a similar capsule and the ash added to the ferric hydrate, after which the whole is heated, gently at first and then to full redness. It is well to cover the capsule at first, to prevent loss by decrepitation of the dry precipitate. After deducting the weight of the filter ash, previously ascertained by burning two or three filters alone, the weight of the iron can be calculated from that of the ignited precipitate, since this consists of Fe_2O_3 and contains 70 per cent. of metallic iron. The assay is sufficiently exact for commercial

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purposes, although the ferric oxide weighed will not be perfectly pure.

XI. ANALYSIS OF COAL.

189. *a. Water* may be determined with sufficient accuracy by drying 150 milligr. of the somewhat coarsely powdered coal in a porcelain capsule or dish over the alcohol flame, at a temperature not high enough to char a bit of filter paper laid on the coal. To avoid mechanical loss the dish may be covered with a watch glass and the coal should be rapidly weighed as soon as cool, repeating the drying until a constant weight is obtained.

b. Coke. The quantity which a coal will yield can be fairly estimated by heating 150 milligr. of the finely powdered coal in a clay crucible, covered with a clay capsule and placed as usual in the square coal, § 170. The crucible is heated gently for a minute or two and is then brought to redness for four or five minutes, after which it is removed from the square coal, allowed to cool while still covered and the coke taken out and weighed. A little covered platinum crucible, Fig. 68, weighing not over two grammes, and provided with a side arm, to keep it upright while on the balance pan, will be very useful for coking. The coke need not be removed from it before weighing.

Fig. 68.

c. Ash. It will be found most convenient to use for this determination a weighed portion of the very finely powdered coke from *b*. This is burned to ash either in a weighed, uncovered platinum capsule, or in an ordinary clay capsule, placed in a square coal as directed for roasting copper assays, etc. From time to time the ash may be stirred with a platinum wire. If it is obtained in a clay capsule it must be removed from this for weighing, and in any case the necessary calculation must be made to allow for only a portion of the coke having been taken. Qualitative tests of the ash can be made, alkalinity pointing to the presence of alkalies or lime; sulphuric acid to the presence of calcium sulphate, originally present as gypsum or formed during the ignition, frequently from presence of iron pyrites and calcite. A reddish or brownish color also indicates the original presence of iron sulphide.

d. Volatile combustible matter. This is obtained by deducting from the original weight of the coal the moisture and the coke.

e. Fixed carbon. This is the weight of the coke less the ash.

f. Absolute heating power, by Berthier's process. Mix 20 milligr. of the finely powdered coal with about 500 milligr. of finely powdered oxychloride of lead in a clay crucible, and cover the mixture with

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as much more of the oxychloride,* upon which is placed eight spoonfuls of powdered glass and finally one spoonful of borax glass as a cover. The crucible is covered with a clay capsule and the whole charge fused in a square coal, as in the lead assay, for eight or ten minutes. One part of carbon reduces thirty-four of lead, hence the heating power of the fuel can be calculated from the weight of the lead reduced.

* Made by fusing together one part by weight of lead chloride and three of litharge in a porcelain crucible, over a Bunsen burner or an alcohol lamp with double draught.

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CHAPTER VIII.

DESCRIPTION OF IMPORTANT ORES AND COALS.

THIS chapter contains descriptions of the minerals usually constituting ores, together with their most distinctive blowpipe characteristics and in some cases their behavior with liquid reagents. For an explanation of certain abbreviations employed, as well as for the mineral formulas, see the explanatory remarks in Chapter IX. All of the true ores naturally occur in massive forms as well as in crystals; some are never crystallized.

Antimony Ores.

190. STIBNITE, *Gray Antimony, Antimony Glance.* IV. Antimony sulphide, Sb_2S_3; Sb=71.8. Crystals usually long columnar, with longitudinal striations, or acicular crystals; also fine radiated or fibrous structure, and often fine-grained and compact. One perfect cleavage direction. Rather brittle; **H.** 2; **G.** 4,6—4,7; lead-gray to blackish; lustre metallic. Fuses very easily and yields in O. F. on coal an odor of sulphur dioxide; with soda gives a strong sulphur reaction and sometimes shows the presence of arsenic by the odor. Yields a white antimony oxide coat.

191. BERTHIERITE. Sulphide of antimony and iron, $FeSb_2S_4$. Sb=57; Fe=13,1. Compact and in fibrous or plumose masses. Brittle; **H.** 2—3; **G.** 4—4,3; dark steel-gray, sometimes inclining to reddish or yellowish-brown; lustre metallic. Fuses, yielding an antimony oxide coat and sulphur dioxide odor, and leaves a dark, magnetic mass, reacting very strongly for iron with borax. It furnishes antimony of inferior quality.

192. *Antimony oxide* (*Stibiconite?*) is said to have been found in large quantities in Sonora, Mexico.

Arsenic Ores.

193. NATIVE ARSENIC, III. (rhombohedral) often containing antimony, and sometimes iron, nickel, cobalt and traces of silver and gold. Usually fine grained, compact and in nodular masses of concentric layers. Brittle; **H.** 3,5; **G.** 5,7—5,8; whitish lead-gray

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on fresh surface, blackening on exposure; lustre metallic. Volatilizes without fusing and behaves in general like arsenic, p. 128, but often leaves a residue, being frequently accompanied by cobalt, bismuth and lead minerals.

194. REALGAR, V. Arsenic Sulphide, AsS; As=70,1. Aurora-red; **H.** 1,5—2; **G.** 3,4—3,6; lustre resinous; transparent to translucent.

195. ORPIMENT, IV. As_2S_3; As=61. Resembles Realgar in general, but is yellow.

196. ARSENOPYRITE, *Mispickel, Arsenical Pyrites.* IV. Arsenide and sulphide of iron, FeSAs; As=46; S=19,6. In short columnar, or tabular crystals; also compact, granular. Brittle; **H.** 5,5—6; **G.** 6—6,2; silver-white to light steel-gray, inclining to yellowish on exposed surfaces; lustre metallic; str. black. In the closed tube yields arsenic and arsenic sulphide; on coal gives sulphur dioxide and arsenical fumes and fuses to a brittle, magnetic globule, which with borax in O. F. shows persistent iron reactions.

Bismuth Ores.

197. NATIVE BISMUTH, III. (rhombohedral), usually containing arsenic and frequently mixed with arsenical silver, lead, cobalt and nickel minerals. Excellent cleavage; generally massive and disseminated. Brittle; **H.** 2,5; **G.** 9,6—9,8; silver-white, with faint reddish tinge, tarnishing brownish or yellowish; lustre metallic. On coal behaves like bismuth, p. 120, but usually gives an arsenical odor. It is the only true ore of bismuth, although much bismuth is obtained by modifications of the processes for smelting ores of lead, silver, etc., when accompanied by various bismuth minerals.

Chromium Ores.

198. CHROMITE, *Chromic Iron.* I. Oxides of chromium and iron, with more or less magnesia and alumina ($\dot{F}e,\dot{M}g)(\ddot{C}r,\ddot{A}l)$; $\dot{C}r_2O_3$=40 to 65. Usually compact, fine-grained and disseminated. Brittle; fracture uneven; **H.** 5,5; **G.** 4,4—4,6; brownish-black; str. brownish to pale grayish-brown; lustre sub-metallic to greasy; occasionally somewhat magnetic. Infusible, or nearly so. Gives a green bead with borax in both flames, fine emerald-green in R. F. The same with S. Ph. Scarcely attacked by acids. The borax bead further treated in O. F. on wire with potassium nitrate becomes yellow, and if powdered, dissolved in water, the solution acidified with

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acetic acid and treated with lead acetate, will yield a yellow precipitate of lead chromate.

Cobalt Ores.

199. SMALTITE. I. Arsenide of cobalt, nickel and iron (Co,Ni, Fe) As_2; Co=3 to 20; Fe=2 to 18; Ni=1 or 2 to 30 or more. Varieties very rich in nickel are called *Chloanthite.* Usually in cubical crystals with octahedral modifications; cleavage octahedral. Generally massive and compact. Brittle; H. 5,5; G. 6,4–7,3; tin-white to light steel-gray; lustre metallic; str. grayish-black. In the closed tube yields metallic arsenic; on coal yields arsenical fumes and fuses to a brittle button, which, in case of typical cobaltic smaltite (*Smaltine*) reacts for cobalt persistently with borax, § 76, *d;* but varieties containing much iron (*Chathamite*) and nickel (*Chloanthite*) show iron and nickel persistently; compare these minerals in the determinative tables, p. 261. The hot nitric acid solution of smaltite, slightly diluted and filtered, does not quickly yield a barium sulphate precipitate on addition of barium chloride solution, although it frequently yields a little at last.

200. COBALTITE, *Glance Cobalt.* I. Sulphide and arsenide of cobalt, $Co(S,As)_2$; Co=35,5; As=45,2; usually contains some iron. Faces of the pentagonal dodecahedron common on crystals; good cubic cleavage in crystals. Brittle; H. 5,5; G. 6–6,1; silver-white, inclining to reddish and steel-gray; lustre metallic; str. grayish-black. Yields no arsenic in the closed tube; on coal fuses, gives arsenical and sulphur dioxide fumes and leaves a brittle button, reacting persistently for cobalt with borax, after possibly first showing a little iron, § 76, *d;* the hot nitric acid solution, slightly diluted and filtered, quickly yields a precipitate with barium chloride solution.

201. LINNAEITE, I, p. 266; Co=58 per cent; octahedral forms predominant; brittle; H. 5,5; G. 4,8–5.

202. ERYTHRITE, *Cobalt Bloom.* V. Hydrous cobalt arsenate, $\dot{C}o_3\ddot{A}s\dot{H}_8$; Co=29,5. In radiated crystals and stellate masses; also earthy. Brittle; H. 2,5; G. 2,9–3; crimson, peach-red, rarely grayish or greenish; str. paler than color; transparent to opaque. Colors the moderately charged borax bead deep blue. Fusible; on coal gives arsenical odor; with the fluxes shows cobalt.

203. ASBOLITE, *Earthy Cobalt.* Hydrous oxides of cobalt and manganese; Co=20 or more; often contains a little iron or copper. Amorphous; H. 1–1,5; G. 2,1–2,2; bluish-black; str. same as color;

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soils paper. Infusible; with borax shows cobalt and more or less manganese.

Copper Ores.

204. NATIVE COPPER, sometimes containing a little silver. I. In crystals, masses, sheets, and wire-like or capillary aggregations; also in disseminated grains. Malleable; H. 2,5–3; G. 8,5–8,9; copper-red, tarnishing yellowish or brownish; lustre and str. metallic; compare copper, p. 115.

205. CHALCOCITE, *Copper Glance, Vitreous Copper*. IV. Copper sulphide, Cu_2S; Cu=79,8. Usually massive; slightly sectile; H. 2,5–3; G. 5,5–5,8; blackish-gray; lustre metallic; str. often somewhat shining. Boils and spirts in O. F. on coal, but yields no magnetic residue. After long treatment in O. F. followed by R. F., especially with addition of soda, yields copper.

206. CHALCOPYRITE, *Copper Pyrites*. II. Sulphide of copper and iron, $CuFeS_2$; Cu=34,6; Fe=30,5. Usually massive; brittle; H. 3,5–4; G. 4,1–4,3; deep brass-yellow when pure; often with a variegated tarnish; str. black with a slight greenish tinge. Fusible, becoming magnetic on coal; in O. F. yields sulphur dioxide odor; when well roasted gives reactions for copper and iron with borax, and on reduction with soda and borax yields metallic copper.

207. BORNITE, *Purple Copper Ore, Erubescite*. I. Sulphide of copper and iron, Cu_3FeS_3 (for crystals); Cu=55 to 70. Usually massive or disseminated. Brittle; H. 3; G. 4,9–5,1; brownish, or purplish copper-red to dark reddish-brown, often with blue and red variegated tarnish; lustre metallic; str. black. Behaves much like chalcopyrite, but sometimes scarcely becomes perceptibly magnetic.

208. TETRAHEDRITE, *Gray Copper, Fahlerz*. I. Sulphide of copper and antimony; Cu=30 to 40, usually; but may be much less; often contains iron, zinc, arsenic, and sometimes mercury, lead or a very little bismuth. It may contain enough silver to constitute a true silver ore. Tetrahedral forms characteristic; also massive and fine-grained. Brittle; H. 3–4; G. 4,4–5,4; Steel-gray to iron-black; str. black; lustre metallic. B.B. fuses, yields sulphurous (and sometimes arsenical) fumes and a coat of antimony oxide; after thorough roasting, copper can be reduced from it, especially on addition of soda. When containing zinc or lead it may yield coats of their oxides also. Any mercury present is obtained by heating with soda in a closed tube. The silver, if present, is detected by cupellation with lead.

209. CUPRITE, *Red Copper, Tile Ore*. I. Cuprous oxide, Cu_2O; Cu

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=88,8; often mixed with hematite. Octahedral forms common; also massive, reticulated or capillary. Brittle; H. 3,5—4; G. 5,7—6; cochineal-red to dark grayish-red, sometimes nearly black on the surface; lustre adamantine to metalloidal-adamantine; also dull; nearly transparent to opaque. B.B. on coal blackens, fuses, and in R. F. finally yields copper.

210. MELACONITE, *Black Copper*. IV. Cupric oxide, Ću; Cu= 79,8. Earthy, massive; also in crystalline scales (*Tenorite*). Dark steel-gray to black; lustre metallic; also dull; H. 3, or less; G. 5,95; on coal fuses and is reduced in R. F.

211. CHRYSOCOLLA. Hydrous copper silicate; ĊuŠiḢ$_2$; Cu= 36, but variable. Black varieties may contain cobalt and nickel. Crypto-crystalline, enamel-like, earthy. Brittle; H. 2—3; G. 2—2,3; green, blue, brown, black; str. greenish-white when pure; lustre feebly vitreous to dull; translucent to opaque. B.B. in O. F. blackens without fusing; with soda in R. F. yields copper; decomposed by HCl, without gelatinizing, and leaves a silica residue.

212. CHALCANTHITE, *Blue Vitriol, Copper Vitriol*. VI. Hydrous copper sulphate, Cu=25.4, is frequently found in water from mines containing copper sulphides, and sometimes in sufficient quantity to admit of profitable separation by precipitation with iron: see p. 269.

213. MALACHITE. V. Hydrous copper carbonate, Ċu$_2$ĊḢ; Cu=57,4. Usually massive; botryoidal, stalactitic and earthy. Brittle; H. 3,5—4; G. 3,7—4,1; color and str. green; translucent to opaque; lustre vitreous to dull. Effervesces with acids; fusible; in R. F. yields copper. It is sometimes mixed with atacamite and then gives a fine blue, copper chloride flame-coloration.

214. AZURITE, *Blue Malachite*. V. Hydrous copper carbonate, Ċu$_3$C̈$_2$Ḣ; Cu=55,2; azure to smalt-blue; otherwise much resembles malachite.

215. ATACAMITE, p. 274, is sometimes found in quantity, especially in Bolivia, S. A. BOURNONITE may constitute at times an important source of copper: see Lead Ores.

Gold Ores.

216. NATIVE GOLD. I. Crystals generally distorted, flattened, elongated; also in nuggets, wire-like, mossy, and in scales or grains, often microscopically small. Malleable; H. 2,5—3; G. 15,6—19,4 (lowest when containing most silver); gold-yellow (to very pale yellow as the silver increases). It may contain 40 per cent. of silver, alloys with 20 to 40 per cent. of silver being often called *Electrum;*

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G. 12,5–15,5. Fusible; soluble in aqua regia, leaving the most of its silver as insoluble chloride, especially after dilution.

217. SYLVANITE, *Graphic Tellurium.* V. Telluride of gold and silver $(Au,Ag)Te_2$; Au=25 to 30; Ag=3 to 15. Often contains lead and antimony, perhaps only as admixtures. Columnar and striated, or lamellar with characteristic, linear markings, whence the name "graphic." Rather brittle; H. 1,5–2; G. 8–8,3; light steel-gray to tin-white, often tinged with yellow; lustre metallic. Fuses, yields tellurium fumes, and finally gives a malleable and usually pale yellow alloy of gold and silver. It may yield a slight yellow coat also.

Petzite, p. 262, is similar in composition, but with more silver.

Iron Ores.

218. HEMATITE, *Specular Iron; Red Hematite.* III. Ferric oxide (sesquioxide of iron) $\dot{F}e_2$; Fe=70. *Specular Iron* is crystalline (rhombohedral); H. 5,5–6,5; G. 5,2–5,3; iron-black to dark steel-gray; str. cherry-red to reddish-brown; lustre metallic; opaque, unless in exceedingly thin plates; often slightly magnetic. A foliated variety is *Micaceous Hematite. Ordinary Hematite* is crypto-crystalline or amorphous; columnar, stalactitic, fibrous, oolitic, or in compact masses and sometimes earthy; H. 3–5; G. 4,5–4,9; blood-red to brownish-red, often tinged with steel-gray; str. red to brownish-red; slightly lustrous to dull. Sometimes compact Hematite is brownish-black, with a jaspery texture, but the streak is always more or less red.

B.B. Hematite in general is infusible or nearly so; in R. F. becomes very magnetic; with borax or S. Ph. reacts only for iron; soluble in strong, hot hydrochloric acid, but sometimes only slowly and in finest powder, earthy varieties often leaving a considerable residue of quartz or other earthy matter. If mixed with Limonite or Turgite it may yield a little water in the closed tube, otherwise none, or but traces.

219. LIMONITE, *Brown Hematite, Bog Iron Ore.* Hydrous sesquioxide of iron, $\dot{F}e_2\dot{H}_3$; Fe=60; but often much less, owing to earthy admixtures; contains 10 to 15 per cent. of water, and often manganese oxides. Amorphous, in radiated, spherical, stalactitic or ordinary compact form; also earthy and oolitic; brittle; H. 5–5,5 (when pure and compact); G. 3,4–4; brown, blackish-brown, yellowish-brown, brownish-yellow (*Ochre*); str. similar in color, but usually somewhat lighter; lustre dull to silky, sometimes sub-metallic, or black and varnish-like on exterior surface; occasionally has

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an iridescent surface. Behaves like Hematite, but yields much water in the closed tube; is also more liable to contain a considerable quantity of phosphorus and sulphur compounds.

Clay-ironstone is partly Hematite or Limonite and partly Siderite. *Ochres*, yellow, brown and red, are impure sesquioxide of iron, soft, earthy and generally behaving like Limonite, but leaving a considerable residue insoluble in HCl.

220. MAGNETITE, *Magnetic Iron Ore.* I. Iron protosesquioxide (ferrous-ferric oxide) $\dot{F}e\ddot{F}e$; Fe=72,4. The octahedron and rhombic dodecahedron are common crystal forms; generally found in coarsely granular or compact masses; also as magnetic sand. Brittle; H. 5,5–6,5; G. 4,9–5,2; iron-black; str. black; lustre metallic, sometimes imperfectly so; opaque; naturally very magnetic and often showing polarity, *i. e.* different parts of a mass attracting different ends of the magnetic needle. Fusible with great difficulty; soluble in hydrochloric acid; with borax or S. Ph. shows only iron. Some so-called *Titanic Iron* belongs here also, especially the titaniferous, magnetic *Iron Sand.*

221. MENACCANITE, *Titanic Iron; Ilmenite; Washingtonite.* Variously regarded as containing ferric and titanic oxides, or as ferrous titanate mixed with ferric oxide. Compare p. 267.

222. FRANKLINITE. I. Oxides of iron, zinc and manganese $(\dot{F}e,\dot{M}n,\dot{Z}n)(\ddot{F}e,\ddot{M}n)$; Fe=46; Zn=10 to 20. Resembles Magnetite in crystalline form and general appearance, but the streak is rather brownish, sometimes reddish-brown. Often feebly magnetic; H. 5.5–6.5; G. 5–5,1; infusible or nearly so; with soda in R. F. a zinc oxide coat; with soda on platinum wire in O. F. gives green sodium manganate, if tested in fine powder and in not too large quantity.

223. SIDERITE, *Spathic Iron.* III. Iron carbonate, $\dot{F}e\dot{C}$; Fe=48,2. Rhombohedral cleavage; also massive, compact and fine-grained, or earthy and impure (Clay Ironstone in part). Brittle; H. 3,5–4,5; G. 3,7–3,9; yellowish-gray, brownish, dark brown when decomposed; str. very pale when pure; lustre vitreous, slightly pearly, or dull when impure; translucent to opaque. B.B. infusible, but blackens and becomes magnetic; effervesces only slightly with cold, but readily with hot acids.

224. PYRITE, *Iron Pyrites.* I. Iron sulphide, FeS_2; Fe=46,7. It is not a true iron ore, but often a valuable source of sulphur. Crystals show especially the faces of the cube, octahedron and pentagonal dodecahedron. Frequently massive; stalactitic; in kidney shaped or spherical masses. Brittle; H. 6–6,5; G. 4,9–5,2; brass-

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yellow, usually light and often with a brown tarnish; lustre metallic; str. brownish-black. In the closed tube yields much sulphur; magnetic after fusion.

Lead Ores.

225. GALENITE, *Galena.* I. Lead sulphide, PbS; Pb=86,6. Cubic cleavage perfect, if any is observed. Generally massive, granular; often finely disseminated. Rather brittle; H. 2,5; G. 7,2–7,6; lead-gray; sometimes with variegated tarnish; str. grayish-black; lustre metallic; fuses easily, yielding a yellow lead oxide coat with some white lead sulphate beyond it; generally contains a little silver, detected by cupelling the reduced lead.

226. BOURNONITE. IV. Lead, copper and antimony sulphides, $PbCuSbS_3$; Pb=42; Cu=13; Sb=25; contains little or no silver. Crystals frequently twinned; massive, fine-grained. Rather brittle; H. 2,5–3; G. 5,7–5,86; steel-gray to nearly black; lustre metallic; str. black. B.B. fuses and yields a yellow lead oxide, and a white antimony oxide coat; the residual button heated in O. F., until it ceases to yield any lead coat, afterward shows copper with borax.

227. CERUSSITE, *White Lead Ore.* IV. Lead carbonate, $\dot{P}b\breve{C}$; Pb=77,6. Crystals often striated and twinned; also massive, granular and compact. Brittle; H. 3–3,5; G. 6,4–6,6 (5,4 in earthy varieties); white, brown, black, rarely green or reddish; lustre adamantine, vitreous, or somewhat resinous; transparent to nearly opaque. On coal fuses and is reduced to lead. Effervesces with nitric acid and dissolves entirely if pure.

228. ANGLESITE, *Lead Vitriol.* IV. Lead sulphate, $\dot{P}b\ddot{S}$; Pb=68,3. Resembles Cerussite in color and lustre; H. 3; G. 6,3; on coal fuses and is reduced to lead; with soda a sulphur reaction; scarcely soluble in dilute nitric acid.

Manganese Ores.

229. PYROLUSITE, *Black Oxide of Manganese.* IV. Manganese dioxide, $\ddot{M}n$; Mn=63.22. Short columnar, or slender divergent crystals; usually in fibrous, radiated, or granular masses; also earthy. Rather brittle to somewhat sectile; H. 2–2,5 or less; G. 4,7–5; dark steel-gray to iron-black; str. black; lustre submetallic to metallic, but often feeble; opaque. Like all the following oxides of manganese, it is infusible, yields strong manganese reactions and dissolves in HCl, evolving chlorine; it may yield a little water in the closed tube.

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230. MANGANITE. IV. Hydrous sesquioxide of manganese, $\dot{M}n\dot{H}$; Mn=62.5. Columnar crystals, with marked longitudinal striations, and often in grouped bundles; one excellent cleavage direction (brachydiagonal) and good prismatic cleavage, at 99°, 40′; also massive, radiated and fibrous; more rarely granular. Rather brittle; H. 3,5—4; G. 4,3—4,4; dark steel-gray to black; str. brown to black; lustre bright, but imperfectly metallic; opaque. Yields much water. It, as well as PSILOMELANE and WAD, are sometimes used for the same purposes as Pyrolusite, but the latter is most valuable.

Mercury Ores.

231. NATIVE MERCURY. Fluid at ordinary temperatures; often contains a little silver; tin-white; G. 13,5—13,6; easily volatilized B.B.

232. CINNABAR. III. Sulphide of mercury, HgS; Hg=86,2. Transparent to nearly opaque; somewhat sectile; H. 2—2,5; G. 8—8,2; cochineal-red, sometimes brownish-red, grayish, or black if impure; str. red; lustre adamantine, occasionally inclining to submetallic; also rather dull in some friable specimens. In the closed tube yields a dark sublimate of mercury sulphide, red when rubbed hard on paper; in the open tube yields sulphurous oxide and when slowly heated a sublimate of mercury; on coal volatilizes entirely and very easily when pure, yielding no perceptible coat, or but a very slight, exceedingly volatile, grayish film of mercury, far from the assay piece. The film rubbed with bright gold colors this silvery-white, through formation of an amalgam; with soda in a closed tube yields mercury. *Hepatic Cinnabar* is an impure variety, mixed with organic matter (Idrialine,) coal and earthy bodies, and is dark red, brown or nearly black, usually with a reddish or brown streak.

Nickel Ores.

233. MILLERITE, *Capillary Pyrites.* III. Nickel sulphide, NiS; Ni=64,45; rarely free from traces of copper, cobalt and iron. Found in acicular crystals and radiated crusts. Brittle; H. 3,5; G. 5,3; brass yellow, often tarnished; lustre and str. metallic; fusible; when roasted shows nickel with the fluxes.

234. NICCOLITE, *Copper Nickel, Arsenical Nickel.* III. Nickel arsenide, NiAs; Ni=43,6. Usually massive, disseminated; brittle; H. 5,5; G. 7,4—7,7; pale copper-red, tarnishing gray or black; str. brownish-black; lustre metallic. In the closed tube yields at most

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a trifling, white, arsenous oxide sublimate; fuses on coal to a brittle button yielding nickel reactions with borax (often also showing cobalt and a little iron); yields arsenical fumes.

235. CHLOANTHITE, and *Chathamite* are varieties of Smaltite, see Cobalt ores, which contain comparatively little cobalt, but much nickel (chloanthite), or iron (chathamite). GERSDORFFITE, *Nickel Glance*, is sometimes an ore of nickel.

236. ANNABERGITE; *Nickel Ochre.* Hydrous nickel arsenate; Ni=29,2, but variable. Microcrystalline; usually encrusting and of earthy texture; H. 2–2,5; G. 3–3,1; greenish to greenish-white; fusible; yields water; with the fluxes shows nickel and usually cobalt also.

237. GARNIERITE. A hydrous silicate containing alumina, iron and magnesia, with varying quantities of nickel oxide, from 2 to 20 per cent., probably due to some disseminated nickel-bearing silicate. It is found in New Caledonia.

Platinum Ore.

238. NATIVE PLATINUM, I; with more or less iridium, rhodium, palladium, osmium, gold and copper, and generally containing 4 to 12 per cent. or more of iron. Usually in small scales or grains; sometimes in large nuggets. Malleable; H. 4–5; G. 17–19; steel-gray, inclining to silver-white; lustre metallic; sometimes magnetic. Infusible B.B., except in *exceedingly* fine wire; soluble only in aqua regia.

Silver Ores.

239. NATIVE SILVER, I; often containing gold, or a little copper, arsenic, antimony and iron. Crystals usually distorted, often greatly elongated octahedra and rhombic dodecahedra; capillary, wire-like, mossy, arborescent; also in sheets and masses, or in disseminated grains. Malleable; H. 2,5; G. 10,1–11; silver-white, tarnishing yellowish, brown or black; fusible, but not easily yielding a marked coat when pure. It is sometimes incrusted with arsenides of iron and cobalt, or is accompanied by various lead or bismuth minerals, all of which cause corresponding blowpipe reactions; it also frequently occurs disseminated in silver chloride (horn silver, cerargyrite).

240. ARQUERITE. I. Silver amalgam; Ag=86,5; Hg=13,5; malleable; H. 2–2,5; G. 10,8; silver-white; in the closed tube yields a mercury sublimate and leaves spongy silver. It constitutes an ore at Arqueros, Chili; while other and brittle silver amalgams, of varying composition, are also found.

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241. ARGENTITE, *Silver Glance.* I. Silver sulphide, Ag_2S; Ag= 87,1. The cube and octahedron are common forms; also massive, wire-like, arborescent, in sheets, crusts and disseminated. Malleable; **H.** 2—2,5; **G.** 7—7,4; blackish-gray to black; lustre metallic, usually not very bright; str. shining. Fusible, swelling or spirting and evolving sulphur dioxide fumes; in O. F. alone finally yields silver, but more easily with soda and borax; may yield a yellowish or brownish coat.

242. PYRARGYRITE, *Antimonial Red Silver, Dark Red Silver Ore, Ruby Silver* (in part). III. Silver and antimony (often also arsenic) sulphides, Ag_3SbS_3; Ag=59,8; Sb=22,5. Massive, disseminated, or incrusting. Rather brittle; **H.** 2—2,5; **G.** 5,75—5,85; dark cochineal-red to blackish-gray; str. cochineal to dark cherry-red; lustre metalloidal-adamantine, sometimes rather dull; translucent to opaque. In the open tube yields sulphur dioxide gas and white antimony oxide fumes (sometimes also arsenous oxide); fuses easily on coal yielding an antimony oxide coat and a sulphur dioxide odor, usually not very strong; sometimes gives arsenical fumes; on longer heating may yield a yellowish, brownish or red coat; is finally reduced to silver, especially with soda and borax.

243. PROUSTITE, *Arsenical Silver Ore, Light Red Silver Ore, Ruby Silver* (*in part*). Silver and arsenic (often also antimony) sulphides, Ag_3AsS_3; Ag=65,46; As=15,15. Resembles Pyrargyrite in crystalline form, mode of occurrence, tenacity and hardness; **G.** 5,5—5,6; bright cochineal-red; lustre adamantine; almost transparent to opaque. In the open tube and on coal yields sulphur dioxide, arsenical (and often also antimonial) fumes; can be finally reduced to silver on coal, as is the case with Pyrargyrite, and may yield a similar colored coat.

244. STEPHANITE, *Brittle Silver Ore, Black Silver.* IV. Silver and antimony sulphides, Ag_5SbS_4; Ag=68,36; Sb=15,44; often contains a little arsenic, iron and copper. Frequently massive, disseminated and incrusting. Rather brittle; **H.** 2—2,5; **G.** 6,2—6,3; iron-black to blackish-gray; lustre metallic; str. black. In the open tube and on coal behaves like Pyrargyrite.

245. TETRAHEDRITE, see Copper Ores, is sometimes very rich in silver, containing from 1 to even 30 per cent. (*Freibergite,*) which is most readily detected by cupellation of the reduced copper and silver button with lead.

246. CERARGYRITE; *Horn Silver.* I. Silver chloride, AgCl; Ag =75,3. Usually massive and disseminated, or incrusting. Somewhat malleable and eminently sectile when pure; **H.** 1—1,5; **G.** 5,6;

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gray, bluish, greenish, but very often brownish on exposure and showing an admixture of native silver when rubbed; lustre, when pure, waxy to feebly adamantine; str. shining; translucent to opaque. Fuses on coal and is finally reduced to silver (very easily with soda;) with copper oxide gives a fine blue color to the inner flame. *Bromyrite* and *Embolite* sometimes constitute silver ores.

247. GALENITE, see Lead Ores, very frequently carries considerable quantities of silver which can be detected by cupellation. CERUSSITE (see Lead Ores) is also sometimes very rich in silver. Other silver minerals, occasionally found in notable quantity, are: *Hessite, Polybasite, Miargyrite, Stromeyerite, Dyscrasite.*

Tin Ores.

248. CASSITERITE, *Tin Stone.* II. Binoxide of tin, SnO_2; Sn = 78,6, but often with several per cent. of sesquioxide of iron. Crystals, stout prisms with pyramidal terminations, very often twinned and showing re-entrant angles; massive, in granular aggregates, or disseminated in almost microscopic grains. *Stream Tin,* in gravel-like masses, of fine-grained texture, and *Wood Tin,* in botryoidal or reniform masses, having concentric structure, internally radiated and compact, are common forms of Cassiterite. Brittle; **H. 6–7**; **G. 6,8–7**; brown, black, yellowish, reddish, gray or nearly white; str. usually very pale, or much lighter than the color of the mineral; lustre adamantine, greasy (rather dull in some Stream Tin); translucent to opaque. Infusible or nearly so, even in the hottest gas flame; usually unaltered B.B. in O. F. in the forceps; in R. F. alone on coal is with some difficulty reduced to metallic tin; quite easily with soda and charcoal dust; mixed with any powdered iron sulphide it yields, in R. F., a copious coat of tin binoxide; not attacked by acids when pure.

249. STANNITE, *Tin Pyrites.* II (or I ?). Sulphide of tin, copper and iron, with more or less zinc; Sn = 25 to 30; Cu, nearly the same; Fe = 6 to 13; Zn = 2 to 10. Usually massive and disseminated, in granular to compact aggregates. Brittle; **H. 4**; **G. 4,3–4,5**; steel-gray, inclining to yellow; str. black; lustre metallic. On coal fuses and yields a tin binoxide coat, sometimes preceded by a rather pure zinc oxide coat; when roasted and reduced with soda and borax, yields a button which, with borax in O. F. on coal gives a bluish glass, or a red glass becoming blue or bluish-green on platinum wire in O. F. Stannite is often mixed with arsenical minerals which cause an arsenical odor B.B. on coal.

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Zinc Ores.

250. SPHALERITE, *Zinc Blende, Black Jack.* I. Zinc sulphide, ZnS; Zn=67, when pure; but generally contains iron sulphide, 20 per cent. in very dark varieties; also frequently cadmium, 1 to 2 per cent. at most; sometimes 1 to 2 per cent. of manganese. Tetrahedral forms common in crystals, which show perfect rhombic dodecahedral cleavage. Massive, in granular, or more rarely fibrous, columnar and radiated aggregates; sometimes in very fine-grained masses, showing no signs of cleavage and resembling some compact limestone in color and texture. Brittle; H. 3,5–4; G. 3,9–4,2; brown, yellowish, black; more rarely grayish, greenish, or colorless; str. colorless, or generally much lighter than the color of the mineral; lustre adamantine to feebly resinous; transparent to opaque. Fusible with difficulty to nearly infusible; in the open tube gives sulphur dioxide; with soda on coal a strong sulphur reaction; roasted and reduced alone, yields a zinc oxide coat, but more easily with soda; the coat is often preceded by a slight cadmium oxide coat; dark varieties roasted yield an iron reaction with borax in O. F. on platinum wire; with HCl evolves sulphuretted hydrogen; may become magnetic B.B. from presence of iron sulphide.

251. ZINCITE, *Red Zinc Ore.* III. Zinc oxide, Żn; Zn=80,3. One excellent cleavage direction; usually massive, in granular or coarsely foliated masses, and disseminated. Brittle; H. 4–4,5; G. 5–5,7; red; str. orange-yellow; lustre somewhat adamantine; more or less translucent. Infusible; on coal in R. F. yields a zinc oxide coat; quietly soluble in acids.

252. WILLEMITE, *Troostite.* III. Zinc silicate, $Żn_2Ši$; Zn=58,1. Massive, fine-grained and disseminated. Brittle; H. 5,5; G. 3,9–4,2; greenish-yellow, green, yellowish, brown, reddish, white; lustre vitreous, inclining to resinous or greasy; str. colorless; transparent to nearly opaque. In general fusible with difficulty to a white enamel; B.B. and with acids behaves much like Calamine, but gives no water. Rarely abundant enough to constitute an ore.

253. CALAMINE, "*Electric Calamine.*" IV. Hydrous zinc silicate, $Żn_2ŠiḢ$; Zn=53,7. Crystals often in fan-shaped aggregates or spherical groups; also fine columnar and fibrous; massive, fine-grained, compact, or earthy. Brittle; H. 5; G. 3,35–3,5; colorless, white, yellowish, brownish; lustre vitreous, pearly, or dull; str. colorless or pale; transparent to nearly opaque. In the closed tube yields water; fusible with great difficulty when pure; on coal in R.

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F. yields a zinc oxide coat, especially with soda; gelatinizes very perfectly with hydrochloric acid; often effervesces when earthy, through admixture with Smithsonite or other carbonates.

254. SMITHSONITE, *"Dry Bone," "Calamine."* III. Zinc carbonate, $\dot{Z}n\breve{C}$; Zn=52; but usually some lime, magnesia, or oxide of iron or manganese replaces part of the zinc oxide; frequently contains cadmium, more rarely a little lead. Massive, granular, earthy, nodular, reniform, stalactitic. Brittle; H. 5; G. 4,1–4,5; colorless, grayish, yellowish, brownish, rarely greenish; str. colorless or pale; lustre vitreous, pearly, or dull; transparent to nearly opaque. Infusible; on coal in R. F. yields a zinc oxide coat, and sometimes a little cadmium oxide; soluble with effervescence in warm dilute acids, dissolving entirely when pure; may yield some water in the closed tube, owing to the presence of earthy impurities, calamine or hydrozincite.

FRANKLINITE; see Iron Ores.

Coals, Lignite and Asphaltum.

255. ANTHRACITE; cannot be kindled by the candle flame; yields more or less water in the closed tube, and perhaps a trifling tarry sublimate; heated B.B. on platinum foil, burns slowly without fusing, leaving a little whitish, grayish, or reddish-brown ash; imparts no color to boiling potassa solution.

BITUMINOUS COAL (*Soft Coal*) and ASPHALTUM; take fire in a candle flame and burn with an empyreumatic odor; in the closed tube yield a brownish or yellowish, tarry liquid; impart little or no color to boiling potassa solution; Asphaltum boiled with ether (*cautiously*, by immersing the test-tube in hot water) colors it wine-red or brownish-red, while Soft Coal colors it at most only pale yellow; Asphaltum fuses quite easily in a candle flame, like sealing wax; some Bituminous Coal does not fuse at all, while some becomes more or less viscid.

LIGNITE (*Brown Coal*); imparts a brown color to boiling potassa solution; B.B. and in the closed tube, resembles some kinds of Bituminous Coal, but often yields much water. The coke from both becomes coated with copper when held between zinc in copper sulphate solution; both may yield a considerable amount of ash.

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CHAPTER IX.

DETERMINATIVE MINERALOGY.

MANY minerals can be determined by chemical tests alone, or by such tests combined with the observation of a few easily recognized physical characters, and the methods of studying certain of these are given in the explanatory remarks below, but an acquaintance with the elements of descriptive crystallography and the meaning of terms used in describing the physical characters of minerals is a necessary preparation for a course in determinative mineralogy. For this the student must consult some standard treatise on mineralogy.

Explanatory Remarks.

1. *Mineral formulas.* In these formulas, as elsewhere, a barred letter in the symbol of an element indicates that two atoms of that element are represented by the symbol, while each of the dots over the symbols represents one atom of oxygen. The small numerals to the right of any symbol multiply not only the number of atoms of the element represented by the symbol, but also the atoms of oxygen indicated by the dots above it. Thus : $\ddot{A}l_3 = 3Al_2O_3$. The formulas can for the most part be readily translated into the empirical formulas now commonly used ; for example : $\dot{C}a\ddot{S} = CaSO_4$ (*Anhydrite*); $\dot{N}a\dot{C}a_4\ddot{S}i_6\dot{H} = Na_2Ca_4Si_6O_{17} + H_2O$, or regarding the hydrogen as basic, two molecules of $HNaCa_2Si_3O_9$ (*Pectolite*) ; $2\,\dot{M}g_3\ddot{B}_4 + MgCl_2 = Mg_7B_{16}Cl_2O_{30}$ (*Boracite*). They have the advantage of often indicating more readily the chemical behavior of minerals.

2. *Systems of Crystallization.* The roman numerals following the names of minerals refer to the systems here enumerated : I. Isometric, or Cubic; II. Tetragonal, or Dimetric ; III. Hexagonal ; IV. Orthorhombic, or Trimetric; V. Monoclinic ; VI. Triclinic.

3. *Cleavage.* The expressions "cleavage in two planes at," or simply "cleavage at," mean that the cleavage planes, sufficiently extended, intersect one another at the angle stated. *Micaceous cleavage*, in consequence of which many minerals can be readily and repeatedly divided into very thin, more or less flexible plates or scales by moderate pressure of the knife-blade against the edges of the plates, is a characteristic of minerals with micaceous structure, and is not to be confounded with fibrous or simply laminated structure.

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4. *Hardness and Tenacity.* The *hardness* of minerals will be stated with reference to the following table of Mohs: 1. *Talc;* 2. *Rock Salt;* 3. *Calcite;* 4. *Fluorite;* 5. *Apatite;* 6. *Orthoclase;* 7. *Quartz;* 8. *Topaz;* 9. *Corundum;* 10. *Diamond.*

The minerals for such a scale should be well crystallized. Gypsum is often substituted for rock salt in the above scale, but in that case the compact, fine-grained variety, alabaster, should be used, since the transparent, crystallized gypsum is too soft.* The harder minerals may be compared with those of the scale by ascertaining which will scratch the other; the softer minerals are best tested by observing the comparative facility with which they and the scale minerals are abraded by a file or scratched by the knife-blade. It is not difficult to form a sufficiently exact estimate of the hardness of most minerals by using the finger-nail, a knife-blade and bits of glass and quartz. Minerals of porous or scaly texture often seem softer than they really are, because the knife-blade easily crushes them, while in fact they may abrade even glass, if rubbed hard against it.

Tenacity may be described in the following terms. *Malleable;* "eminently" so, when the mineral can be hammered quite thin without cracking. *Brittle* minerals fly off in powder, more or less fine, when scratched. *Sectile* minerals have also different degrees of tenacity; "eminently" sectile minerals can be cut like lead; others, like molybdenite, pare off in little shavings when cut, but are not quite malleable; while between the quite sectile and the quite brittle minerals there are many which crumble readily under the hammer, but cut rather like a soft lead pencil; to such the terms "somewhat sectile" or "rather brittle" may be applied.

5. *Lustre and Color.* Lustre is due to the light reflected from a mineral. "*Metallic lustre*" is sufficiently described by its name; when necessary, it should be observed on a fresh surface of the mineral, which will always be absolutely opaque. "*Sub-metallic*" or "*imperfectly metallic lustre*" is not always so easily recognized, but it is often an important characteristic. It is suggestive of, while not the same as metallic lustre, and the powder of minerals possessing it may even be earthy in appearance. The term will be confined in the following tables to minerals which are opaque even on the thinnest edges, while their very fine powder or streak will be dark colored (not necessarily black), with but one or two exceptions, and those of very rare minerals. Minerals whose lustre may be truly doubtful will be found both under minerals with sub-metallic and with non-metallic lustre. "*Non-metallic lustre,*" which is not confined to transparent minerals, has no semblance of metallic lustre, although certain minerals share in some degree the lustre of both classes, having "metalloidal-pearly," "metalloidal-adamantine" lustre, etc.

Color is highly characteristic in the case of minerals with metallic lustre and generally also with those having sub-metallic lustre, but much less so in most cases of non-metallic lustre, although there are prevailing colors for many of

* Nicol, *Elements of Mineralogy,* Edinburgh, 1873, gives the following substitute for the above scale of hardness: Minerals scratched by the finger nail have hardness 2.5 or less; those scratching copper, 3 or more; polished white iron has hardness 4; window glass, 5 to 5.5; a steel point or file, 6 to 7; quartz (crystals or flint) 7. In testing for hardness, streak and even fusibility and magnetism, great care must be taken, to avoid forming an erroneous opinion through presence of minute impurities in minerals.

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these. The color of an opaque mineral must be observed on a fresh fracture if the mineral is liable to alter superficially.

6. *Streak.* The very fine powder of a mineral has the same color as its "streak," which is best observed for practical purposes, by rubbing the mineral on hard, well-burned, unglazed porcelain, such as the streak-tables sold for this purpose. The lustre of the streak may be important; "metallic" streaks are like those made by a coin; "shining" streaks are not necessarily metallic.

7. *Magnetism and Pyroelectricity.* For practice on the commoner minerals, a good, small bar magnet will suffice. The particles to be tested may be placed on very smooth paper if necessary, and mere adhesion of the particles to the magnet must not be mistaken for true attraction. For universal work a delicate magnetic needle is used, in which case it is generally advisable to use rather larger fragments of minerals. Pyroelectric minerals when heated develop a power of attraction, which may be tested by means of a deer's hair or cotton fibres.

8. *Action of acids.* For *gelatinization* see § 112, *d*. Other acids than hydrochloric are to be used when specified. To determine in doubtful cases whether a mineral has been much decomposed by an acid, after being boiled with it for fifteen minutes or so, in very fine powder, dilute the acid slightly with water, filter the liquid (or decant it if it settles clear enough) and add a slight excess of ammonia, followed by addition of sodium phosphate solution, if necessary. Should only a trifling precipitate form, the mineral cannot have been largely decomposed. If care is taken to use only moderate quantities of water and reagents, the test can generally be made in a watch-glass over the water bath.

Carbonates must leave little if any residue when appropriately treated with acids, since a slight effervescence, ceasing before the mineral is all dissolved, or evidently not proportionate to the quantity of the mineral taken, only indicates the accidental presence of some carbonate.

9. *Ignition-loss.* This is due in general to the expulsion of water* and is best estimated by strongly igniting the mineral in a very light platinum crucible; a blast-lamp being necessary with some minerals, especially magnesian silicates. With fragments weighing about 0.5 grm. the loss may be determined on the Jolly spring-balance, Fig. 69.

10. *Specific gravity.* This is conveniently determined on the spring-balance, Fig. 69. The spiral wire spring *b*, set by the movable rod *C*, carries two little pans, *c* and *d*, the lower of which is kept *constantly* beneath the water in the glass on the movable arm *B*, without ever resting on the bottom of the vessel. The position of the index *m* is first read off from the scale *A*, engraved on a mirror, so that the index *m* and its reflected image may fall in the same line of vision to avoid parallax. The mineral fragment, 0.5 to 2 grm. (a spiral spring of corresponding strength being used) is then placed in the pan *c* and another reading taken, after which the fragment is placed in the pan *d*, and a final reading taken. Calling these readings *x*, *y*, *z*, in the order in which they were taken, the specific gravity is found thus: sp. gr. $= \frac{y-x}{y-z}$, no matter what may be the actual weight of the fragment.

* It may here be stated that many minerals which contain no water when pure will sometimes yield a little water in the closed tube, owing to incipient alteration or other causes. Judgment and experience alone will enable the student to form a correct opinion as to the result of this very important test.

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11. *Fusibility and volatility.* Before deciding that a mineral is infusible the finest splinters must be tested; * coarser ones serve better to determine the degree of fusibility. If not otherwise specified, the fusibility of minerals should be tested in the forceps with the O. F., fresh pieces of the minerals being used. As a matter of precaution it may be well, with minerals of metallic lustre, first to try the fusibility on coal, since any mineral that does not fuse easily on coal may, with care, be safely tested in the forceps. Merely swelling, becoming cloudy or otherwise changing in appearance, does not necessarily indicate fusion. Consult also § 40.

Volatility is best determined by the use of quite small fragments and mere mechanical action, such as flying into fine powder, must not be mistaken for true vaporization of the mineral. When volatile minerals leave a *residue of impurities* some judgment becomes necessary. The impurities will often be earthy and generally infusible at the comparatively low temperature which suffices to volatilize the pure mineral.

12. *Method of using the Determinative Tables.* Only pure minerals with well defined characteristics should be tried at first; afterwards the student may learn to judge whether a reaction is due to any slight impurity by its relative intensity. Thus, a few minute grains of iron pyrites, accidentally present, may cause a slight brown stain on the silver in the test for sulphur, § 103, *c*, but if sulphur was an important constituent of the mineral, the powder of a fragment about as large as a hemp seed should cause a dark stain a half an inch or so in diameter, if the test is well made.

The examination of a mineral should always start at the beginning of Part I., Classifying Examination, p. 255, the properties and behavior of the mineral being successively compared with the descriptions there given in order, following class, division, section and group, until the tests have brought the student to a point where the Detailed Examination, Part II., is to be taken up, on the page to which reference may have been made in the Classifying Examination.

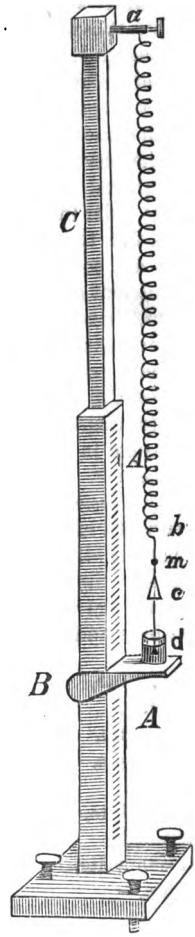

Fig. 69.

Suppose *Pyrrhotite* is in hand. Its metallic lustre places it in Class I.; its brittleness in Division B; it yields neither an arsenical odor nor a coat, therefore we pass to Section II., p. 256; the sulphur dioxide odor ob-

* The term "infusible" should be regarded as a relative one. In the following tables minerals which cannot be rounded on the thinnest edges of very fine splinters, with either an oil or a gas O. F., will be called infusible; other minerals which can be so fused, although generally not by beginners, will be called infusible or nearly so; those which can be fused even by the beginner, if careful, will be called fusible on thin edges, fusible with some difficulty, easily fusible, etc., according to their nature; or their fusibility will be compared with the scale, p. 48.

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tained on coal in O. F. places it in Group I., and the page reference in bold type, at the right hand of page 256, indicates that the detailed examination of the minerals in this group will be found on page 265. Since the mineral does not correspond to the minerals under 1, it is tested as directed in 2, and as it yields neither silver nor copper, it cannot belong under *a*, so that the test with borax is next applied, as directed in *b*. This test furnishes a yellow to red bead while hot, becoming lighter yellow on cooling, therefore the mineral belongs under a, and being naturally magnetic is found to correspond to *Pyrrhotite*, since *Pentlandite*, with which alone it could be confounded, shows nickel abundantly in the reduced bead.

When several minerals are enumerated in the same subdivision the reactions of the mineral in hand should be compared with the descriptions of those minerals in the order in which they are named. Thus:—in the subdivision 2, of Group V, p. 267, the mineral should first be compared with *Franklinite* then with *Chromite*, and so on, until a mineral is found to which it corresponds.

Different specimens of certain minerals may vary so much in composition that it sometimes becomes necessary to introduce the names of those minerals into subdivisions in which the minerals would not ordinarily belong. In such cases the names of the minerals are prefaced with the word "compare," indicating that the detailed behavior of those minerals must also be studied, by reference to the proper subdivision. For instance, on p. 277, *Epidote*, being often magnetic after treatment in R. F., would then also come into the section of minerals becoming magnetic.*

13. *Minerals for Practice.* Some of the following minerals have been already mentioned in the list on p. 3. They have been selected with the purpose of introducing all of the more important tests, for the commoner elements especially, and after the student has learned to determine them accurately, he can determine any mineral which can be identified by blowpipe analysis, aided by a few simple wet tests and decided physical characteristics.

Alabandite,	Chromite,	Menaccanite,
Allanite,	Cinnabar,	Millerite,
Almandite,	Cobaltite,	Molybdenite,
Amphibole,	Columbite,	Muscovite,
Apatite,	Cryolite,	Natrolite,
Apophyllite,	Cuprite,	Niccolite,
Argentite,	Epidote,	Opal,
Arsenopyrite,	Fluorite,	Orthoclase,
Barite,	Franklinite,	Prehnite,
Beryl,	Graphite,	Pyrargyrite,
Bismuth,	Gypsum,	Pyrite,
Bournonite,	Halite,	Pyrolusite,
Calamine,	Hematite,	Pyromorphite,
Calcite,	Kalinite,	Pyroxene,
Cassiterite,	Labradorite,	Quartz,
Chalcocite,	Lepidolite,	Realgar,
Chalcopyrite,	Magnesite,	Rhodochrosite,

* In making such comparisons it is necessary to ascertain first whether the mineral in hand gives general reactions that place it in the same group, etc., as the comparison mineral.

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Ripidolite,	Stibnite,	Triphyllite,
Rutile,	Strontianite,	Ulexite,
Siderite,	Sylvanite,	Uraninite,
Smaltite,	Talc,	Vivianite,
Smithsonite,	Tiemannite,	Wolframite,
Sphalerite,	Topaz,	Wollastonite,
Spodumene,	Tourmaline,	Zircon.

In the following tables the names of the commoner minerals and of such of the rarer ones as present marked characteristics are printed in capitals, or bold type, and the student should at first confine his attention to them.

14. *Abbreviations used in the Tables.*

amorph. .	amorphous.	gryh. . . .	grayish.
blkh. . .	blackish.	**H.** . . .	hardness.
brnh. . .	brownish.	HCl. . . .	Hydrochloric acid.
clvg. . .	cleavage.	HNO_3. . .	Nitric acid.
diff. . .	with difficulty.	H_2SO_4. . .	Sulphuric acid.
fus. . .	fusibility, or fuses.	infus. . . .	infusible.
G. . . .	specific gravity.	ppt. . . .	precipitate.
gelat. . .	gelatinous, or gelatinizes.	rdh. . . .	reddish.
grnh. . .	greenish.	str. . . .	streak.

PART I.

CLASSIFYING EXAMINATION.

Paragraphs in square brackets [ ] may be omitted when using the tables only for practice. Page references in bold type are to the detailed examination in Part II.

CLASS I. MINERALS WITH METALLIC OR SUB-METALLIC LUSTRE.

DIVISION A. Malleable and eminently sectile (or fluid); hardness not above 5. . . . . . p. **260**

DIVISION B. Not both malleable and eminently sectile.

SECTION I. Yield a decided arsenical odor, or a definite coat of some kind (not necessarily arsenical) by the preliminary tests.*

GROUP I. Yield an arsenical odor (and perhaps some coat) alone on coal in R. F., ... p. **260**

* *Preliminary tests.* Heat a small fragment alone on coal in R. F., at first gently, afterwards strongly, noting any odor and coat, and finally using a strong O. F., if no arsenical odor or decidedly colored coat has already been obtained. If the mineral is easily and completely volatile, yielding no odor or marked coat, compare it with **Cinnabar**, p. 265; if it proves infusible on coal and yields no arsenical odor, pass to Section II, p. 265; in all other cases proceed regularly with the examination as indicated above.

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GROUP II. Alone in R. F. yield a coat which colors the inner flame bright green (tellurium), or greenish-blue (tellurium and selenium). The mineral shows tellurium, on coal with porcelain, etc., § 101, *d*, . . . p. 262

GROUP III. Alone in R. F. yield a strong selenium odor; the mineral colors the flame blue or green, but any coat fortaed colors it blue, . . p. 262

GROUP IV. Yield a white coat, which tinges the inner flame pale yellowish-green (antimony.) * The coat from certain silver minerals may finally become red,† § 38, *b*, ... p. 263

GROUP V. Fuse and give a coat, which is decidedly yellow (lead or bismuth) even after cooling. It may be accompanied by a considerable white coat, p. 264

GROUP VI. Minerals not included in the foregoing groups, . p. 265

SECTION II. Yield neither an arsenical odor nor the coats ‡ of Section I.

GROUP I. Alone in O. F. on coal yield sulphur dioxide.§ . . p. 265

GROUP II. [Yield silver buttons; fusible; hardness not above 3; silver-white], ... p. 266

GROUP III. With soda and borax on coal in R. F. yield one or more copper buttons; or yield tin, ... p. 266

GROUP IV. With S. Ph. in O. F. on platinum wire give, at least when in considerable quantity, a violet-red bead (manganese); all infusible, . p. 266

GROUP V. Become magnetic ‖ in R. F., ... p. 267

GROUP VI. Minerals not included in the preceding groups, . . p. 267

* When experience in observing the pale yellowish-green flame-coloration from the antimony coat, § 96, *a*, and § 42, is lacking, the powdered mineral may be heated with the alcohol flame in a short open tube, about 45 millim. long, the white fumes issuing from the upper end of the tube being afterwards allowed to escape against the base of the blue alcohol flame, to produce the antimony flame-coloration. If necessary the upper part of the tube may be heated, to volatilize the antimony oxide sublimate in it; but the test on coal, properly made, is sufficient for antimony in this case.

† After long heating a few of the minerals in this group may yield a decided *zinc oxide* coat, yellowish while hot, but nearly or quite white on cooling; no decided yellow coat (*lead* or *bismuth oxide*) will form, although the silver minerals in this group may yield, in addition to the white antimony oxide coat, a yellowish-brown or brown coat; they are easily identified, in doubtful cases, by the comparatively large silver button which they finally yield (see p. 257, fourth foot-note).

‡ Only Molybdenite, Sphalerite and Sternbergite, p. 265, and the Amalgams and Cassiterite, p. 266, may yield coats, and even they not always readily.

§ The open tube, § 32, 1, *a*, may be used to obtain the sulphur dioxide, but if the finely powdered mineral is spread out on the coal, brought to full redness in O. F. and quickly held under the nose, the sulphurous odor can easily be detected; at least in all of the commoner minerals, except perhaps *Molybdenite*. The very volatile Cinnabar requires the open tube.

‖ If the mineral does not become magnetic after long heating alone in R. F. on coal, treat it with R. F. in the forceps; sometimes it may be necessary to heat it very strongly within the point of the blue cone of a good O. F., when only the bar magnet is used to test the magnetism. The heated portion is broken into fine grains, not powder, and then tested with the magnet, unless it shows magnetic properties without being crushed.

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CLASS II. MINERALS WITHOUT METALLIC OR SUB-METALLIC LUSTRE.

DIVISION A. Having taste,* sometimes feeble; hardness not above 3; volatile or fusible on coal, although some may finally leave an infusible residue.

1. Effervesce briskly with cold dilute nitric acid; react alkaline with test-papers, ... p. 268
2. Fuse easily in O. F. to a clear bead ;† yield a boron reaction, alone or with Turner's flux, § 113, *a*, ... p. 268
3. Deflagrate on coal (nitrates, § 110, *a*), . . . p. 268
4. Give a strong chlorine reaction, § 104, *a*, 1 ; show little or no sulphur, § 103, *c*, ... p. 269
5. Yield with soda a strong sulphur reaction, . . . p. 269

DIVISION B. Minerals without taste.

SECTION I. Readily and completely volatile on coal ;‡ hardness not above 3, ... p. 270

SECTION II. Yield a malleable metal or some definite coat with soda and borax in R. F. on coal.¶

GROUP I. Yield silver, § 83 ; all fusible on coal : or yield tin, § 94, *a;* the mineral being infusible, ... p. 270

GROUP II. Yield bismuth or lead buttons ; [also a yellow lead or bismuth oxide coat, at least after longer blowing, and often without soda or borax ; may also yield some white coat and arsenic odor ‖], . . . p. 271

GROUP III. [Yield a cadmium coat, but no metal: or an antimony coat ; sometimes also metallic antimony], ... p. 272

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* This division does not include minerals yielding an arsenical odor with soda in R. F. on coal.

† If quite impure the cold bead may not be clear.

‡ Use a gentle O. F. at first, afterwards a stronger one ; the minerals of this section yield *only a white coat*, unless too rapidly heated (§ 35, *a*, *note*) ; antimony minerals may yield at first a residue of metallic globules, but they are volatilized quite easily and, in any event, yield only a white coat. Compare p. 253, remarks on volatility.

¶ Treat the finely powdered mineral in a proper cavity, § 50, *a*, using a powerful R. F. if necessary, and bringing the fluxes together with the spatula, if they do not melt into one mass. From time to time note any odor or coat produced. If buttons of metal are obtained, §§ 50, 51, cleanse and test them as to malleability, color, and behavior on coal, at first in a gentle O. F., then in a stronger one. Should the metal be non-volatile, yielding no considerable yellow coat (*lead* or *bismuth*), or white one close to the button (*tin*), and yet have no characteristic silver or copper color, fuse it alone on coal in O. F. for a short time and then beside borax, to purify it. (The silver minerals of this section yield buttons which may at first give a decided white, reddish, or yellowish coat, but on heating the metal for some time in O. F. and then testing it on a fresh coal it will yield little if any coat in a moderate O. F., while tin, lead, or bismuth, will yield abundant coats so long as any metal remains.) The above preliminary tests are used to group the minerals as above.

‖ The only difficultly fusible mineral in this group is *Plumbogummite.*

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GROUP IV. Yield a zinc coat, § 79, *a*, *b*, ... p. 272
GROUP V. Yield copper, ... p. 273

SECTION III. Alone in R. F. on coal, yield an arsenic odor, or a magnetic residue, or both.*

GROUP I. Yield an arsenic odor and sometimes a magnetic residue also; fuse below 5, ... p. 274
GROUP II. Yield a magnetic residue, but no arsenic odor, . . p. 274

SECTION IV. Minerals not included in the preceding sections.

SUB-SECTION I. After strong ignition alone react alkaline ;† hardness below 5, ... p. 278

SUB-SECTION II. Yield one of the following special reactions.

GROUP I. Yield a lithium flame :‡ streak pale or colorless, . p. 279
GROUP II. Yield reactions for boric or phosphoric acid.¶

1. React for boric acid;¶ all fuse rather easily except *Warwickite*, p. 279, and some varieties of *Tourmaline*, p. 285, ... p. 279
2. React for phosphoric acid,¶ . . . . . p. 280

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* Test first on coal; if the mineral is not magnetic after cooling, treat it alternately in O. F. and R. F. for some time, ending with a good R. F.; see also the last foot-note, p. 256.

† Ignite in the forceps unless too fusible. The mineral, after cooling, is placed on moistened turmeric paper in a watch-glass, when the alkaline reaction will be shown by a brown or reddish-brown spot, which soon forms, at least immediately under the fragment. Minerals are not to be powdered for this test, unless they decrepitate very strongly, for some minerals not belonging here will give an alkaline reaction otherwise.

‡ Use the bisulphate of potassium mixture, § 60, in case of doubt.

¶ *a*. The careful student may well employ only the flame-coloration test (more or less distinctly green flame) when working on the minerals *for practice alone*. The finely powdered mineral is tested on wire with sulphuric acid for the flame-coloration, unless a green flame obtained in the forceps alone renders the acid unnecessary. If no green flame is obtained in either way, pass to Group III.; otherwise, make first a test for boric acid. as in *b*, below (but only the test with turmeric paper for confirming the presence of boric acid, as there directed). If boric acid is found, pass to p. 279 for the special examination; if not, make one of the tests for phosphoric acid, in *c*, below. Should neither boric nor phosphoric acid be found, the flame-coloration is probably due to traces of copper and the mineral does not belong here. **Tourmaline** will not be found here if only sulphuric acid was used for the flame test, but it will be found elsewhere.

b. For *universal work* test first with Turner's flux, § 113, *a*; if a green flame is obtained, confirm the presence of boric acid with turmeric paper, § 113, *d*, 2, after fusion with soda, etc. Should no boric acid be found, pass to *c*, below, and also test for phosphoric acid as there directed, even if Turner's flux has failed to produce a green flame.

c. The simplest universal test for phosphoric acid is with magnesium, § 109, *b*, but the most convenient laboratory test is usually that with ammonium molybdate, applied at once to the nitric acid solution of the (commoner) minerals, or employed after decomposition with the flux, § 109, *c*, in case of minerals not attacked by nitric acid. If phosphoric acid is found, pass to p. 280, for the detailed examination.

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GROUP III. Yield with borax in O. F. on wire a decided manganese reaction,* p. 281

GROUP IV. Yield with S. Ph. on wire reactions as follows :—†

1. In O. F. a blue or violet bead, becoming blue in R. F.; streak brown or black, p. 282

2. In O. F. a yellow bead (cold); unchanged in R. F., . . p. 282

3. In O. F. a colorless, reddish, yellowish or greenish bead (cold), which assumes a decided color in R. F., while the mineral gives one of certain special reactions, p. 282

GROUP V. Yield a sulphur reaction with soda on coal; the reaction may not be strong in case of some of the silicates belonging here, . . p. 283

[SUB-SECTION III. Minerals not included in the preceding Sub-Sections.

Note. When using the tables only for practice, pass to Sub-Section IV., p. 291.

GROUP I. Fusibility from 1 to 5.

1. Gelatinize perfectly with hydrochloric acid, ... p. 283

2. Decomposed by hydrochloric acid with separation of silica, but without gelatinizing perfectly, ... p. 284

3. Soluble in hydrochloric acid, without separation of silica, . . p. 285

4. Almost or entirely unaffected by hydrochloric acid, . . . p. 285

GROUP II. Infusible or fusible above 5.

1. Ignited, moistened with cobalt solution and again ignited, assume a fine blue color,‡ p. 287

2. Entirely or almost entirely soluble in hydrochloric acid (or nitric, if necessary) without gelatinizing or leaving any considerable residue of silica, . . p. 288

3. Decomposed by hydrochloric acid, either gelatinizing or leaving a residue of silica, p. 288

4. Minerals not included above], ... p. 289

SUB-SECTION IV. Minerals not included in Sub-Sections I and II. ¶

Note. Here are included only minerals for practice; all of them will also be found in Sub-Section III.

A. Structure eminently micaceous, ... p. 291

B. Structure not micaceous.

GROUP I. Yield little or no water.

1. Fusibility not above 5, ... p. 292

2. Fusibility above 5, ... p. 293

GROUP II. Yield water.

1. Fusibility not above 5, ... p. 295

2. Fusibility above 5, ... p. 296

* Very small quantities produce a decided amethyst-red color; larger ones cause an intense red or even opaque bead. Minerals yielding a sulphur dioxide odor in O. F. on coal must be very thoroughly roasted.

† The minerals should be very finely powdered, since some are but slowly attacked; the silicates may leave a considerable silica skeleton, while the cold beads are in some cases opalescent.

‡ Hard minerals must be finely powdered before the ignition.

¶ For *Coals, Lignite* and *Asphaltum*, see p. 249.

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PART II.

DETAILED EXAMINATION.

CLASS I. MINERALS WITH METALLIC OR SUBMETALLIC LUSTRE.

Division A. Malleable and eminently sectile (or fluid); hardness not above 5.

Section I. Fusible (or fluid).

Mercury, § 231. SILVER, § 239. *Electrum*, § 216. GOLD, § 216. COPPER, § 204. *Lead;* lead-gray; **H.** 1,5; slowly volatilized B.B., giving a yellow coat. *Maldonite*, Au_2Bi: pinkish-white to silver-white; gives a yellow coat and finally leaves a gold button. *Chilenite*, $Ag_{10}Bi$?; silver-white; gives a yellow coat and finally a silver button. *Arquerite*, § 240.

ARGENTITE, § 241: *Acanthite*, IV, is a silver sulphide dimorphous with Argentite, and similar in behavior and appearance: *Jalpaite* is Argentite with about 14 % of copper; **G.** 6,88. Compare *Hessite* and *Whitneyite*.

Section II. Infusible.

Platinum, § 238. *Palladium*, I; light steel-gray; **H.** 4,5; **G.** 11,8 –12,2; slowly soluble in nitric acid to a red solution. *Allopalladium*, III; a dimorphous form of Palladium. *Iron*, I; steel-gray to iron-black; **H.** 4,5; **G.** 7–7,8; very magnetic.

Division B. Not both malleable and eminently sectile.

Section I. Yield a decided arsenical odor or a definite coat.

Group I. Yield an arsenical odor (and perhaps some coat) alone in R. F. on coal.

1. *Yield an arsenical odor and may give a white, grayish, or slight brownish coat.**

a. Entirely and readily volatile: ARSENIC, § 193. *Allemontite*, $SbAs_3$ (but variable); tin-white to light-gray; fuses easily; yields arsenical and antimonial fumes on coal. Compare ANTIMONY, sometimes arsenical, p. 263.

b. Not entirely volatile: fusible in R. F. on coal, except some *Lölingite*, p. 261.

* The coat may finally become reddish in case of silver minerals. The silver minerals belonging here may also give a yellowish coat, but they are distinctly characterized by soon yielding silver globules; compare the foot-note to section II., p. 257.

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a. Well roasted and reduced with soda and borax, the following yield copper; all except the last three yield a strong sulphur reaction with soda. (Compare POLYBASITE, in b, which yields cupriferous silver; also *arsenical* TETRAHEDRITE, p. 263): *Tennantite*, I, $(Cu,Fe)_4As_2S_7$; and *Epigenite*, IV, Cu,Fe,As,S, yield magnetic residues alone: compare *Carrollite*. *Enargite*, IV, Cu_3As,S_4; str. black; and *Binnite*, I, $Cu_6As_4S_9$; str. reddish-brown; yield little or no silver by cupellation. *Algodonite*, Cu_6As; Cu = 83,5; steel-gray to white; tough; *Domeykite*, Cu_3As; Cu = 71,7; white; and *Whitneyite*, Cu_9As; Cu = 88,4; bronze to rdh-white; somewhat malleable.

b. With soda and borax the following yield silver; also a sulphur reaction, § 103, *c*: POLYBASITE $(Ag_2Cu)_9(Sb,As)_2S_{12}$; black; str. black; with borax on coal in O. F. shows copper. PYRARGYRITE, § 242.

c. Treated as in § 76, *d*, the following yield a persistent cobalt reaction (perhaps after first showing a little iron); the residual button is brittle: SMALTITE, § 199; *Skutterudite*, I, $CoAs_3$, clvg. cubic; *Glaucodot*, IV, $(Co,Fe)(S,As)_2$; all tin-white to gryh.-white; yield metallic arsenic if strongly heated in the closed tube; the hot nitric acid solution of Glauc., diluted slightly and filtered, quickly yields a ppt. with barium chloride solution. Compare CHLOANTHITE (no ppt. with barium chloride), *Chathamite* and *Cobaltic Arsenopyrite*, in d and e, following.

COBALTITE, § 200, like Glaucodot, but no arsenic sublimate in the closed tube; compare *Ferro-cobaltite*, in e.

d. Treated as under c, above, yield a persistent nickel reaction: *Corynite*, I, Ni,Sb,As,S, G. 6, and *Wolfachite*, IV, composition similar, show antimony reactions; compare *Ullmannite*. GERSDORFFITE, NiAsS; white to steel-gray; the hot nitric acid solution, slightly diluted and filtered, quickly gives a ppt. with barium chloride; in the closed tube a sublimate of arsenic sulphide. NICCOLITE, § 234, does not give the ppt. with barium chloride and yields at most only a faint white sublimate. CHLOANTHITE, I, $NiAs_2$ (Co,Fe); tin-white; may show considerable cobalt at first with borax; yields metallic arsenic in the closed tube. *Rammelsbergite*, IV; $NiAs_2$; tin-white, sometimes reddish; also yields arsenic. Compare *Chathamite*, below.

e. Treated as under c, above, yield a persistent iron reaction: ARSENOPYRITE, § 196; LÖLINGITE (*Leucopyrite*), IV, $FeAs_2$; sometimes scarcely fusible on coal; and *Chathamite*, I; all silver-white to light steel-gray; yield metallic arsenic in closed tube (Ar-

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senopyrite, carefully heated, also gives a more or less distinct, yellowish to brownish, arsenic sulphide sublimate at first); Chathamite finally shows nickel and cobalt with borax. *Cobaltic Arsenopyrite* (including *Danaite*), behaves like Arsenopyrite, but finally shows considerable cobalt with borax. *Ferro-cobaltite* at first shows much iron, but finally behaves like Cobaltite, in c.

2. *Arsenical odor; coat distinctly yellow in part at least; all fusible on coal.*

a. Yield a bismuth reaction, § 90, *b*: *Rionite;* Cu,Bi,As,Sb,S; gives copper reactions. *Alloclasite;* Fe,Co,Bi,As,S; with borax shows iron and cobalt. Compare BISMUTH, § 197, often impure.

b. Yield a lead reaction, § 86, *b*: *Sartorite,* IV; $PbAs_2S_4$; bluish-gray; str. rdh.-brown; decrepitates strongly: *Dufrenoysite,* IV; $Pb_2As_2S_5$; dark lead-gray; str. rdh.-brown: *Jordanite,* IV; Pb, As,S; lead-gray; str. black. Compare *Geocronite,* p. 264; *arsenical Tetrahedrite,* p. 263; *Galenite,* § 225, (only when quite impure).

Group II. Alone in R. F. yield a coat which colors the inner flame bright green or greenish-blue; also yield the tellurium reaction with porcelain, etc., § 101, d; all fusible on coal. (Some of the minerals afford a slight selenium odor.)

1. *Tellurium;* tin-white; very fusible and volatile.

2. *With potassium iodide and sulphur yield no very marked yellow or red coat:*

Hessite, Ag_2Te; lead-gray; with soda a silver button. SYLVANITE, § 217: *Petzite,* steel-gray to iron-black, is similar, but has Ag= 40 to 46 %. *Melonite,* Ni_2Te_3; rdh.-white; with borax shows nickel.

3. *With potassium iodide and sulphur show lead or bismuth:*

TETRADYMITE, Bi_2Te_2S; Bi=50 to 60 %; steel-gray, soft; *Joseite* is similar, but has about 78 % Bi. *Altaite,* PbTe; tin-white, inclining to yellowish; no silver, or only traces. *Nagyagite;* mainly Au,Pb,Te; blkh.-gray; soft; yields gold with little silver. *Müllerite* (plumbiferous Sylvanite); silver-white to brass-yellow; yields gold containing much silver. SYLVANITE, § 217, may yield a slight yellow or red coat with the iodide mixture.

Group III. Yield in R. F. a strong selenium odor; color the flame blue or green; any coat which forms colors the flame blue; fusible on coal.

1. *Crookesite;* Cu,Tl,Ag,Se; gives a green flame and leaves a copper button containing a little silver.

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2. *With soda give yellow coats:*

Frenzelite, Bi_2Se_3; with potassium iodide and sulphur shows bismuth. *Lehrbachite;* Pb,Hg,Se; with soda in the closed tube yields mercury. *Zorgite;* Cu,Pb,Se; in O. F. gives a copper button. CLAUSTHALITE, Pb,Se. Compare *Naumannite,* below.

3. *Not included above:*

Guadalcazarite, $HgZnS_7$, with very little Se; with soda in the closed tube yields mercury; on coal, a zinc coat: TIEMANNITE, Hg_6Se_5; readily volatile; in the closed tube with soda gives mercury. *Berzelianite,* Cu_2Se; roasted and reduced with soda, yields copper: *Eucairite,* Cu,Ag,Se; similarly treated leaves an alloy of copper and silver: *Naumannite,* Ag_2Se?; finally leaves silver; often contains lead.

Group IV. Fuse and yield a white coat, which tinges the inner flame pale yellowish-green and shows antimony reactions by § 96, b. (The coat from certain silver minerals may be finally red.)

1. *Fuse and volatilize entirely* (use small fragments): ANTIMONY, str. and color tin-white; perfect clvg.; H. 3,5; G. 6,2; no sulphur dioxide odor on coal. STIBNITE, § 190.

2. *Not entirely volatile:*

a. Silver-white: *Dyscrasite* (antimonial silver), Ag_2Sb; alone yields finally a silver button.

b. Light copper-red: *Breithauptite,* NiSb; the residual button finally shows nickel with borax.

c. Lead-gray to black; sulphur dioxide odor on coal; strong sulphur reaction with soda:

a. After long treatment alone on coal, in alternate O. F. and R.F., leave a silver button (which may need purifying by treatment with soda in R. F., followed by short treatment with borax in O. F.): STEPHANITE, § 244. *Miargyrite,* V, $AgSbS_2$; black to blkh.-gray; str. dark red; Ag=36% nearly, Compare POLYBASITE, p. 261; PYRARGYRITE, § 242.

b. Heated for some time with a strong O. F. and R. F. alternately, the residue powdered and thoroughly roasted, two or three times if necessary, and finally reduced with soda, yield copper (which may need to be purified by alloying it with its volume of lead and treating it with boric acid, § 88, *f,* 2): TETRAHEDRITE, § 208; sometimes *arsenical: Stylotypite,* similar, yields a magnetic button alone on coal: *Spaniolite* is mercuriferous Tetrahedrite: *Chalcostibite,* IV, $CuSbS_2$, behaves much like Tetrahedrite.

c. Not included above: *Ullmannite,* I, Ni,Sb,S,As; G. 6,3;

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with borax, finally shows nickel persistently. BERTHIERITE, § 191.

Group V. Fuse and yield a coat which is decidedly yellow (lead or bismuth) in part at least, even after cooling.

1. *With potassium iodide and sulphur show bismuth,* § 90, *b:*

a. The button obtained with R. F. alone is heated for some time in O. F.; should it not then evidently contain copper, it is alloyed with a little lead and treated with boric acid for copper, § 88, *f*, 2 (if the button treated alone volatilizes entirely, copper is naturally absent):

a. Yield no copper, or only traces: BISMUTH, § 197. The three following yield strong sulphur reactions with soda. BISMUTHINITE, Bi_2S_3; light lead-gray, inclining to tin-white or yellowish, str. gray: KOBELLITE, Pb_3BiSbS_6; dark lead-gray, str. black; antimony reactions: *Chiviatite,* $Pb_2Bi_6S_{11}$; lead-gray; contains 2.5 % Cu.

b. Yield a considerable residue of copper: *Aikinite,* $PbCuBiS_3$; lead-gray to steel-gray; **G. 6,75**: *Emplectite,* $CuBiS_2$; tin-white; **G. 5,1—5,2**: Wittichenite, Cu_3BiS_3; dark steel-gray to lead-gray; **G. 4,3—4,5**: compare *Tetrahedrite,* § 208, which rarely contains a little bismuth.

2. *With potassium iodide and sulphur show lead,* § 86, *b; all yield sulphur dioxide on coal, or give a strong sulphur reaction with soda.*

a. The button obtained with R. F. as in 1, *a,* and further treated as there directed, yields no copper, or only traces; it may leave a silver button (but only when it fails to be volatilized alone in O. F.):

a. The coat from the raw mineral gives a strong antimony flame* and shows antimony with tin and S. Ph.; the mineral contains little or no silver: *Jamesonite,* $Pb_2Sb_2S_5$; *Plagionite,* $Pb_4Sb_6S_{13}$; BOULANGERITE, $Pb_3Sb_2S_6$; *Meneghinite,* $Pb_4Sb_2S_7$; *Geocronite,* $Pb_5Sb_2S_8$; *Zinkenite,* $PbSb_2S_4$: the foregoing are not easily distinguished B.B. alone; Geocronite is often arsenical; *Jamesonite,* IV, has basal cleavage; *Plagionite,* V, has not.

b. As in **a**, but contain much silver: *Freieslebenite,* V., $(Pb,Ag_2)_5Sb_4S_{11}$: *Diaphorite,* IV, is dimorphous with it. *Brongniardite,* I; $PbAg_2Sb_2S_5$. *Polytelite,* Pb=38.3; Ag=5.78; Sb=22.4; Zn=6.8; S=22.5; Cu=0.3.

c. The coat from the raw mineral shows no antimony reactions: GALENITE, § 225: *Huascolite* is probably a mixture of galenite and sphalerite.

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* The open tube may be used if necessary; see first foot-note, p. 256.

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b. The button obtained as in 1, *a*, yields copper with boric acid: *Castillite;* Pb,Cu,Ag,Fe,Zn,S; copper-red; the coat shows zinc. *Cuproplumbite* is probably galenite containing chalcocite. BOURNONITE, § 226. *Tetrahedrite* (*plumbiferous*).

Group VI. Not included in the foregoing groups.

1. Fusible, yielding a coat which is nearly white, when cold, and with cobalt solution becomes green: STANNITE, § 249: compare SPHALERITE, below.

2. Certain mercury minerals may yield a slight and very volatile grayish coat:

Amalgam, p. 245, leaves a silver residue. CINNABAR, § 232, and *Metacinnabarite* volatilize entirely.

Section II. Yield neither an arsenical odor, nor the coats of Section I.

Group I. Yield sulphur dioxide fumes.

Compare the easily volatile CINNABAR, § 232, and *Metacinnabarite.*

1. *The following yield special reactions:*

MOLYBDENITE, MoS_2; bluish lead-gray; very soft; infusible; in the forceps gives a green flame; gives a molybdenum reaction, § 99, *f;* in O. F. may give a white, blue and reddish coat.

SPHALERITE, § 250, gives with soda a zinc coat and strong sulphur reaction.

2. *The mineral is well roasted alone and then tested in separate portions; the minerals in 2 fuse below 5:*

a. One portion is reduced with soda and borax (two volumes of soda and one-half volume of borax) and the resulting slag is gently hammered between paper, unless it contains metallic buttons that can be at once picked out. If the button does not appear to be pure copper or silver it is purified by treatment beside borax in O. F.:*

a. The button is silver: *Sternbergite,* $AgFe_2S_3$; alone in R. F. gives a magnetic button.

b. The button is malleable copper, or with borax in O. F. on coal yields strong and persistent copper reactions: the following five

* If the mineral shows no blue flame with hydrochloric acid on coal, § 88, *a*, save time by passing to *b*, unless testing for Sternbergite also; if it shows a blue flame, the tests in *a* and b must be made, in order to prove the presence of considerable copper, since *traces* of copper do not bring the mineral into the division b, but may cause the blue flame.

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yield alone on coal in a strong O. F., followed by R. F., magnetic buttons, but Bornite may become but very slightly magnetic : CHALCOPYRITE, § 206; *Cubanite,* I, $CuFe_2S_3$, bronze-yellow, cubical clvg.; *Barnhardtite,* (Cu, about 47 %, Fe,S), massive ; BORNITE, § 207 ; *Carrollite,* $CuCo_2S_4$, with the fluxes shows cobalt and copper (contains a little arsenic).

CHALCOCITE, § 205, does not become magnetic.

b. A portion is tested with borax on platinum wire in a pure and long continued O. F.

a. The bead is red to yellow while hot, lighter yellow or brownish-yellow on cooling (greenish, if containing a little copper as an impurity). In R. F. the decidedly yellow bead becomes bottle-green, or nearly black (iron) : PYRRHOTITE, III; Fe_7S_8 to $Fe_{11}S_{12}$; often contains a little Ni ; dark bronze-yellow ; naturally magnetic ; H. 3,5—4,5. PYRITE, § 224. *Marcasite,* IV, light gryh.-yellow to grnh.-yellow, is dimorphous with Pyrite and behaves similarly. *Pentlandite,* Fe_2NiS_3, bronze-yellow ; the reduced borax bead yields abundant scales of metallic nickel ; often contains a little copper ; acts on the magnetic needle before fusion.

b. The bead shows manganese, nickel, or cobalt : ALABANDITE, I, MnS ; str. dirty-green, shows manganese ; *Hauerite,* I, MnS_2, str. brnh.-red, does the same, but yields a sulphur sublimate in the closed tube. MILLERITE, § 223, and *Beyrichite,* Ni_5S_7, show nickel ; Beyrichite is lead-gray and yields a sulphur sublimate. LINNAEITE, I, Co_3S_4, reddish silver-white, gives a blue bead in both O. F. and R. F., but the variety *Siegenite* may contain 40 % of nickel.

Group II. Yield Silver.

Amalgam ; Ag,Hg ; Ag=26 to 36 % ; brittle : *Arquerite,* Ag_{12} Hg ; malleable : both are silver-white and yield mercury sublimates.

Group III. With soda and borax on coal in R. F. yield copper or tin.

CASSITERITE, § 248, yields tin. The following yield copper. CUPRITE, § 209. *Crednerite,* $\dot{C}u_3\ddot{M}n_2$; black ; fus. diff. ; with borax in O. F. a violet bead. MELACONITE (*Black Copper, Tenorite*), § 210, Ċu ; steel-gray to black, sometimes dull and earthy. Compare *Rabdionite.*

Group IV. With S. Ph. in O. F. on wire show manganese; all infusible.

1. *Yield little or no water :*

PYROLUSITE, § 229 ; M̈n. *Polianite ;* light steel-gray ; H. 6,5—7,

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is a variety of Pyrolusite. *Braunite,* II, $\dot{M}n$; brnh.-black; str. black; **H. 6–6,5.** *Hausmannite,* II, $\dot{M}n\dot{M}n$; iron-black; str. brown; **H. 5–5,5;** good basal clvg. Compare PSILOMELANE, below.

2. *Yield water:*

MANGANITE, § 230. PSILOMELANE, $\dot{M}n$ (sometimes $\dot{B}a, \dot{K}, \dot{H}$;) massive, stalactitic, botryoidal; iron-black to bluish-black; lustre sometimes sub-metallic; **H. 5,5–6;** no clvg: *Lithiophorite,* similar (with a little $\dot{C}u, \dot{C}o, \ddot{A}l, \dot{L}i$), gives a lithia flame; **H. 3–3,5.** Compare PYROLUSITE, just above.

Group V. Become magnetic in R. F.

1. *Gelatinize with hydrochloric acid; all fuse below* 5:

ALLANITE, $\dot{C}a, \dot{C}e, \dot{L}a, \dot{D}i, \dot{F}e, \ddot{A}l, \dot{S}i$; B.B. intumesces strongly; the HCl solution, after separation of silica, yields with ammonia an abundant ppt. which leaves a white residue when warmed with enough oxalic acid; this residue ignited, treated with HCl, filtered out and again ignited, yields brownish or brick-red cerium oxide. ILVAITE, $\dot{C}a_2\dot{F}e_4\ddot{F}e\ddot{S}i_4$; intumesces slightly. *Fayalite,* $\dot{F}e_2\ddot{S}i$; brown to greenish-black; and *Hortonolite,* $\dot{M}g, \dot{F}e, \ddot{S}i$; yellow to black; str. gryh.-white; have both crystalline clvg.

2. *Do not gelatinize with hydrochloric acid.*

FRANKLINITE, § 222. CHROMITE, § 198. WOLFRAMITE, V; $(\dot{M}n, \dot{F}e)\ddot{W}$; black to brnh.-black; str. rdh.-brown to blkh.-brown; **H. 5–5,5; G. 7,1–7,5;** fus. 3; dissolved in S. Ph. and reduced on coal beside tin, gives tungsten reactions, §100, *d.* MENACCANITE, III; $\dot{F}e\dot{T}i$; but some $\ddot{F}e(\dot{M}g, \dot{M}n)$; black, inclining often to brownish, also steel-gray; str. black to dark brnh.-red; infus. or nearly so; two or three well charged S. Ph. beads, treated with strong HCl and tin, show titanium, § 69, *d.* (The beads must be *finely* powdered.) *Iserite* (*iron sand*), probably belongs here in part.

MAGNETITE, p. 242. *Magnesioferrite,* $\dot{M}g\ddot{F}e$; and *Jacobsite,* $(\dot{M}g, \dot{M}n)(\ddot{F}e, \ddot{M}n)$ resemble Magnetite, but the HCl solution of Magnesioferrite shows magnesium, while Jacobsite also gives manganese reactions.

HEMATITE, § 218. LIMONITE, § 219. TURGITE, $\ddot{F}e_2\dot{H}$; rdh.-black; str. red; behaves much like Limonite; decrepitates in the closed tube.

Compare some of the minerals in group VI.

Group VI. Minerals not included in the preceding groups.

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Compare CHROMITE and WOLFRAMITE, in the preceding group.

GRAPHITE, C (with more or less Fe, Si and earthy impurities); black to gray; foliated, cleavable or granular-massive; very soft; infus.; soils paper.

Samarskite, Ce, Y, Zr, Th, U, Fe, Cb(W); velvet-black; lustre bright sub-metallic to greasy; str. dark rdh.-brown; fuses on edges; shows columbium, § 70, *b*, and sometimes a little tungsten. *Yttrotantalite*, Ca, Y, Fe, U, Ta, Cb(little W); yellow to black; sub-metallic to greasy; str. grayish; infus., but whitens or becomes pale yellowish.

URANINITE, UU_2; greenish to gryh.-black; infus. or nearly so; shows uranium with borax and S. Ph., Table VI, p. 91; the HNO_3 solution yields a yellow precipitate with ammonia. *Perofskite*, I, CaTi; gryh.-black to black; infus.; shows titanium reactions: Compare *Rutile* and *Brookite*, p. 283.

COLUMBITE, IV; FeCb(Ta); black; G. 5,4—6,4; one rather distinct cleavage direction; infus. or nearly so; shows columbium, § 70, *b*; sometimes a little tungsten, also, Table VII, p. 133. *Tantalite*, G. 7—8, no clvg., quite similar in general character.

Fergusonite, II, $Y_3Cb(Ta)$; black; clvg. pyramidal, if any; columbium reaction; G. 5,8. Compare *Polycrase*, *Aeschynite*.

Iridosmine; Ir, Os, Rd, Ru; tin-white; osmium reactions; includes *Sysserskite*, G. 21,1, and *Newjanskite*, G. 19,4.

CLASS II. MINERALS WITHOUT METALLIC OR SUB-METALLIC LUSTRE.

Division A. Having taste; hardness not above 3; volatile or fusible on coal; may finally leave an infusible residue.

1. *Effervesce with dilute acid; reaction alkaline:*

Natron, $NaCH_{10}$; effloresces on exposure; liquefies when gently heated: *Thermonatrite*, NaCH; effloresces on exposure: *Trona*, $Na_2C_3H_4$.

2. *Fuse to beads clear when cold; reactions for boric acid*, § 113, *a*:

Sassolite, BH_3; feebly acid reaction: BORAX, NaB_2H_{10}; feebly alkaline reaction.

3. *Deflagrate on coal:*

NITRE, KN; strong potassa flame, at least through cobalt glass: *Soda Nitre*, NaN: *Nitro-calcite*, CaNH; leaves a lime residue; deflagrates feebly.

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4. *Give reactions for chlorine; contain little or no sulphur:*

Sal Ammoniac, NH_4Cl; volatilized without fusing: SYLVITE, KCl; a strong potassa flame; when pure leaves finally no residue on coal: HALITE (*Common Salt*) NaCl; also yields little or no residue after long heating; gives a strong soda flame: *Carnallite,* $KMgCl_3\dot{H}_6$; leaves a magnesia residue: *Tachydrite,* $CaMg_2Cl_6\dot{H}_{12}$; the aqueous solution yields a ppt. with ammonium chloride and carbonate.

5. *Yield a strong sulphur reaction with soda,* § 103, *c:*

a. Heated with potassa solution yield ammonia: *Mascagnite,* $(NH_4)_2SO_4$; entirely volatile: *Tschermigite,* $NH_4Al(SO_4)_2+12\,H_2O$; leaves an alumina residue.

b. The following, ignited for a short time on coal, leave an alkaline residue:

Aphthitalite, $\dot{K}\ddot{S}$, giving a potassa flame; *Mirabilite,* $\dot{N}a\ddot{S}\dot{H}_{10}$, yielding much water; and *Thenardite,* $\dot{N}a\ddot{S}$, leave little or no residue after long ignition on coal.

Polyhalite, $\dot{K}\dot{C}a_2\dot{M}g\ddot{S}_4\dot{H}_2$, giving a potassa flame through cobalt glass, and *Glauberite,* $\dot{N}a\dot{C}a\ddot{S}_2$, when dissolved in hot dilute HCl, yield no ppt. with a slight excess of ammonia, but afterwards an abundant one with ammonium oxalate.

The alkaline residue from the following six minerals, strongly ignited for some time in O. F. on coal, afterwards shows magnesia when heated with cobalt solution: *Picromerite,* $\dot{K}\dot{M}g\ddot{S}_2\dot{H}_6$, gives a potassa flame, at least through cobalt glass; *Kainite,* $KCl\dot{M}g\ddot{S}\dot{H}_3$, is similar, but also gives chlorine reactions: EPSOMITE, $\dot{M}g\ddot{S}\dot{H}_7$; $H_2O=48$ to 51; easily soluble in water: *Kieserite,* $\dot{M}g\ddot{S}\dot{H}$; $H_2O=13$; slowly soluble; taste bitter-saline and very feeble, unless the mineral is altered: similar minerals are *Loeweite,* $\dot{N}a_2\dot{M}g_2\ddot{S}_4\dot{H}_5$; $H_2O=14{,}6$; taste feeble; and *Bloedite* (*Simonyite*) $\dot{N}a\dot{M}g\ddot{S}_2\dot{H}_4$; $H_2O=21.5$; very soluble.

KALINITE (*Potash Alum*) $\dot{K}\ddot{A}l\ddot{S}_4\dot{H}_{24}$; the strongly ignited residue shows alumina with cobalt solution.

c. The residue from the following minerals is not alkaline:

Goslarite, $\dot{Z}n\ddot{S}\dot{H}_7$; the strongly ignited residue becomes green on ignition with cobalt solution: *Alunogen,* $\ddot{A}l\ddot{S}_3\dot{H}_{18}$; the residue becomes blue with cobalt solution.

Morenosite, $\dot{N}i\ddot{S}\dot{H}_7$; the residue gives nickel reactions with borax: *Bieberite,* $\dot{C}o\ddot{S}\dot{H}_7$; the residue shows cobalt reactions: *Chalcanthite,* $\dot{C}u\ddot{S}\dot{H}_5$; the residue yields, with soda in R. F., copper:

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Fauserite, (Ṁg,Mn,S̈,Ḣ) ; the residue yields strong manganese reactions.

Iron Sulphates ; the residue gives strong iron reactions with borax, and on coal in R. F. becomes very magnetic ; here are included—*Pettkoite* (Ḟe,F̈e,S̈,Ḣ) ; $H_2O=1,5$; yielding little water in the closed tube ; and the following, yielding much water : Melanterite, V, $FeS\dot{H}_7$, vitriol-green and wholly soluble in water when pure : *Coquimbite,* III, $F\ddot{e}S_3\dot{H}_9$, yellowish and wholly soluble in cold water : *Roemerite,* yielding a faint zinc coat with soda and borax : *Fibroferrite,* yellow, fibrous, silky, taste weak, partly soluble : *Botryogen,* yellow to red, partly soluble : *Voltaite,* I, dark green to black.

Division B. Minerals without taste.

Section I. Volatile (yielding no colored coat).

1. Sulphur ; takes fire easily and burns freely when removed from the flame, yielding a strong sulphur dioxide odor.

2. *With soda in R. F. yield an arsenical odor ; the coat from the mineral alone shows no antimony flame or reactions.*

Realgar, § 194, aurora-red. Orpiment, § 195, yellow. *Claudetite,* IV ; and *Arsenolite,* I, are both white Ä̇s.

3. *The coat from the mineral alone shows antimony reactions.*

Kermesite, $Sb_2S_3+Sb_2O_3$; cherry-red. *Valentinite,* IV ; and *Senarmontite,* I, are white or colorless, fusible S̈b. Compare *Cervantite, Stibiconite, Volgerite,* p. 272.

4. *Yield a white or grayish coat, or no coat :*

Calomel, Hg_2Cl_2, whitish ; and Cinnabar, § 232 ; both yield a mercury sublimate with soda, § 84, *a : Metacinnabarite* is a gryh-black variety of Cinnabar ; str. black. Compare *Cotunnite.*

Section II. Yield a malleable metal or a definite coat with soda and borax in R. F.

Group I. Yield silver, all being fusible on coal ; or yield tin, the mineral being infusible.

1. *Yield silver :*

Proustite, § 243 : *Xanthoconite,* III, $Ag_9As_3S_{10}$, is similar, but has color and str. pomegranate-yellow to yellowish-brown. Pyrargyrite, § 242 ; compare *Miargyrite,* p. 263. Cerargyrite, § 246 ; *Bromyrite,* AgBr ; *Iodyrite,* AgI ; and *Embolite* Ag,Br,Cl, are eminently sectile : consult §§ 104, 105, 106, as to their behavior with potassium bisulphate.

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2. *Yield tin:*

CASSITERITE, § 248. (Certain columbates, tantalates, and other minerals yield traces of tin.)

Group II. Yield lead or bismuth.

1. *Yield a strong arsenical odor:*

a. Show bismuth, § 90, *b*: *Walpurgite*, $\ddot{B}i_5\ddot{U}\ddot{A}s_2\ddot{H}_{10}$, gives uranium reactions. Compare the bismuth minerals in 2, *a*; they sometimes contain arsenic.

b. With potassium iodide and sulphur show lead (a yellow coat, perhaps partly white, but with no red): *Bayldonite*; $\dot{P}b,\dot{C}u,\ddot{A}s,\ddot{H}$; yields copper. MIMETITE, $3\dot{P}b_3\ddot{A}s+PbCl_2$; gently fused in the forceps in O. F., becomes irregularly crystalline on cooling; on coal is finally completely reduced to lead: *Campylite* and *Hedyphane* (calciferous) are varieties containing $\ddot{P}$: compare PYROMORPHITE, p. 272. *Araeoxene*; $\dot{P}b,\dot{Z}n,\ddot{A}s,\ddot{V}$; zinc reaction. Several minerals in 2, *b*, may contain arsenic.

2. *No arsenical odor; with potassium iodide and sulphur show bismuth or lead:*

a. Show bismuth: *Pucherite*, $\ddot{B}i\ddot{V}$; green bead with S. Ph. in R. F. on coal. *Bismutite*, $\ddot{B}i,\ddot{C},\ddot{H}$; effervesces with HCl. *Eulytite*, $\ddot{B}i_2\ddot{S}i_3$; gelat. with HCl. *Bismite*, $\ddot{B}i$.

b. Show lead:

a. Yield copper, § 88, *f*, 2: *Vauquelinite*, $\dot{C}u\dot{P}b_2\ddot{C}r_2$; with nitre shows chromium, see Crocoite. *Linarite*, $\dot{P}b\ddot{S}\dot{C}u\ddot{H}$; with soda a sulphur reaction. *Percylite*, Pb,Cu,Cl,O; a copper chloride flame.

b. Yield definite reactions or colored beads with S. Ph. as follows: WULFENITE, $\dot{P}b\ddot{M}o$; the moderately charged bead in O. F. is nearly or quite colorless and shows molybdenum, § 99, *f*. *Stolzite*, $\dot{P}b\ddot{W}$; shows tungsten, § 100, *d*. CROCOITE, V; $\dot{P}b\ddot{C}r$; hyacinth-red; str. orange; S. Ph. bead green in O. F. and R. F.; fused on wire with nitre in O. F., and the mass heated with water, gives a yellow solution, which yields a yellow precipitate when acidified with acetic acid, at least after addition of lead acetate: *Phoenicocroite*, IV; $\dot{P}b_3\ddot{C}r_2$; behaves much like Crocoite; str. brick-red. *Vanadinite*, III, $3\dot{P}b_3\ddot{V}+PbCl_2$; yellow, brown, red; S. Ph. bead in O. F. yellow to yellowish-green, in R. F. green; shows chlorine, § 104, *a*, 1: *Descloizite*, IV; $\dot{P}b_2\ddot{V}$; olive-green, brown, black; similar, but may show no chlorine: *Dechenite*; $\dot{P}b,\dot{Z}n,\ddot{V}$; similar to the foregoing; yields zinc reaction.

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c. Yield certain special reactions: PYROMORPHITE, $3\dot{P}b_3\ddot{P}+PbCl_2$; fuses easily on coal, but is not reduced to lead, unless containing some arsenate (compare Mimetite, 1, *b*); the fused bead is usually very distinctly but irregularly crystalline on cooling; the nitric acid solution shows phosphoric acid, § 109, *c*. *Plumbogummite;* $\dot{P}b,\ddot{A}l,\ddot{P},\dot{H}$; swells, but does not fuse; yields phosphoric acid reactions; with cobalt solution shows alumina. *Nadorite,* $\dot{P}b\ddot{S}b+PbCl_2$, gives antimony and chlorine reactions. *Bindheimite,* $\dot{P}b,\ddot{S}b,\dot{H}$; yields water and antimony reactions.

d. With dilute HNO_3 yield carbon dioxide; some are not completely soluble: CERUSSITE, § 227. *Phosgenite,* $\dot{P}b\ddot{C}+PbCl_2$; shows chlorine. *Leadhillite,* IV; $3\dot{P}b\ddot{C}+\dot{P}b\ddot{S}$; partly soluble; with soda a sulphur reaction: *Susannite,* III, behaves similarly and is perhaps only a twinned form. Compare *Lanarkite,* below.

e. With soda give a strong sulphur reaction: ANGLESITE, § 228. *Lanarkite,* V; $\dot{P}b\ddot{S}+\dot{P}b$ (sometimes $\dot{P}b\ddot{C}$); perfect basal clvg.

f. Not included above: *Minium,* $\dot{P}b\ddot{P}b$; red. *Massicot,* $\dot{P}b$; yellow. *Matlockite,* II; $\dot{P}bPbCl_2$; G. 7,2; clvg. imperfect; *Mendipite,* IV; $\dot{P}b_2PbCl_2$; G. 7; perfect prismatic clvg.; and *Cotunnite,* IV; $PbCl_2$; G. 5.2; very easily volatilized: all show chlorine reactions.

Group III. Yield a cadmium or antimony coat.

Greenockite, CdS, yields a cadmium coat; infus.; yellow to brown.

The following yield antimony coats: *Cervantite,* $\ddot{S}b\ddot{S}b$, infus. B.B. alone on coal: *Stibiconite,* $\ddot{S}b\ddot{S}b\dot{H}_2$, yielding 5 % of water, and *Volgerite,* $\ddot{S}b\dot{H}_5,H_2O=15$, are similar: *Stibioferrite* ($\dot{F}e,\ddot{S}b,\dot{H}$), alone on coal gives a magnetic residue.

Group IV. Yield a zinc coat.

1. *Adamite,* $\dot{Z}n_4\ddot{A}s\dot{H}$; fus.; strong arsenical odor.

2. *With soda a sulphur reaction; with HCl in powder yield hydrogen sulphide:* SPHALERITE, § 250: *Danalite,* $3(\dot{B}e,\dot{F}e,\dot{M}n)_2\ddot{S}i+Zn\,S$; gelat. with HCl.

3. *With HCl effervesce strongly* (*give carbon dioxide*), which is best identified by using dilute nitric acid on a fresh portion, § 111, *a*. *Aurichalcite,* $\dot{C}u_2\dot{Z}n_3\ddot{C}_2\dot{H}_3$, yields copper: SMITHSONITE, § 254: *Hydrozincite,* $\dot{Z}n_3\ddot{C}\dot{H}_2$, is similar, but yields much water. Compare CALAMINE, which gelatinizes, § 253; also *Fowlerite.*

4. *Gelatinize with HCl:* CALAMINE, § 253. WILLEMITE, § 252. *Roepperite,* $(\dot{M}g,\dot{F}e,\dot{M}n,\dot{Z}n)_2\ddot{S}i$; dark green to black; fus. diff.;

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three cleavage planes at right angles with one another. Compare *Tephroite*, which may contain disseminated zinc minerals.

5. *Not included above: Jeffersonite* (var. of Pyroxene); grnh-black; fus. 3: ZINCITE, § 251: GAHNITE, $\dot{Z}n\ddot{A}l$; infus.; H. 7,5–8; str. grayish or greenish. Compare *Fowlerite*, p. 282, and FRANKLINITE, § 222.

Group V. Yield copper.

1. *Yield an arsenical odor; colors more or less distinctly green: Chenevixite;* $\ddot{F}e,\dot{C}u,\ddot{A}s,\dot{H}$; in R. F. alone gives a magnetic scoria.

The following resemble one another in general behavior B.B. OLIVENITE, IV; $\dot{C}u_4\ddot{A}s(\ddot{P})\dot{H}$; olive to blkh-green; fused for a moment in O. F. in the forceps, gives a dark, crystalline mass on cooling; $H_2O=3.8\ \%$: EUCHROITE, IV; $\dot{C}u_4\ddot{A}s\dot{H}_7$; emerald to leek-green; $H_2O=19\ \%$: *Clinoclasite*, V; $\dot{C}u_6\ddot{A}s\dot{H}_3$; dark bluish-green; $H_2O=7.5\ \%$: *Tyrolite;* $\dot{C}u,\ddot{A}s,\dot{H}$ (with intermixed $\dot{C}a\check{C}$); effervesces with HNO_3: *Chalcophyllite*, III; $\dot{C}u,\ddot{A}l,\ddot{A}s,\dot{H}$; decrepitates strongly; H. 2; G. 2,4–2,6: *Erinite;* $\dot{C}u,(\ddot{A}l),\ddot{A}s,\dot{H}$; minutely crystalline structure; decrepitates strongly; H. 4,5–5; G. 4; $H_2O=5\ \%$: *Liroconite*, V; $\dot{C}u,\ddot{A}l,\ddot{A}s,\ddot{P},\dot{H}$; sky-blue to verdigris-green; $H_2O=$ 22 to 25 %; the HNO_3 solution yields a white precipitate (alumina) with excess of ammonia: *Cornwallite;* $\dot{C}u,\ddot{A}s,\dot{H}$; amorphous; $H_2O=13\ \%$; G. 4,2: compare *Libethenite*, p. 274; *Torbernite*, below.

2. *Effervesce strongly with hydrochloric acid (yielding carbon dioxide):* MALACHITE, § 213, AZURITE, § 214. *Atlasite* $\dot{C}u,\check{C},CuCl_2\dot{H}$; copper chloride flame. Compare MELACONITE, § 210, which may contain carbonates.

3. *Decomposed by HCl with separation of silica.** CHRYSOCOLLA, § 211. DIOPTASE, $\dot{C}u\check{S}i\dot{H}$; green; gelat. *Cyanochalcite;* blue; probably impure Chrysocolla; yields phosphoric acid reaction.

4. *Not included above:*

a. Red, or dark gray to black; str. dark gray, black or reddish; soluble in strong HCl: CUPRITE, § 209. MELACONITE, § 210: *Lampadite*, is similar, but shows much manganese. *Rabdionite;* $\dot{M}n,\ddot{M}n,\ddot{F}e,\dot{C}u,\dot{C}o,\dot{H}$; alone on coal gives a magnetic globule; reacts also for cobalt and manganese.

b. With S. Ph. in O. F. give a yellowish-green to green bead; also give certain special reactions: *Torbernite,* $\ddot{U}_2\dot{C}u\ddot{P}\dot{H}_8(\ddot{A}s)$;

* Or yield a silica skeleton with S. Ph.

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yields phosphoric acid reactions; the HNO_3 solution gives a bluish-green precipitate with excess of ammonia. *Volborthite,* $\dot{C}u_4\ddot{V}\dot{H}$ (usually some $\dot{C}a$); yields vanadium reactions, § 81.

c. The HNO_3 solution shows phosphoric acid by treatment with ammonium molybdate (other tests for phosphoric acid may be used if more convenient): PSEUDOMALACHITE; $\dot{C}u,\ddot{P},\dot{H}$; G. 4–4,4; H_2O=6.3 to 9%. LIBETHENITE, $\dot{C}u_4\ddot{P}\dot{H}$; G. 3,6–3,8; H_2O=3.8%. *Tagilite,* $\dot{C}u_4\ddot{P}\dot{H}_3$; G. 4; H_2O=10.5 %. Compare *Turquois,* which may contain a few per cent. of copper.

d. Give an intense sky-blue color to the flame: ATACAMITE, $3\,\dot{C}u\dot{H}+CuCl_2$; green; str. apple-green; yields water; *Tallingite,* blue to green, str. white, composition and behavior similar. *Nantokite,* Cu_2Cl_2; white.

e. Yield a sulphur reaction with soda: BROCHANTITE, $\dot{C}u_4\ddot{S}\dot{H}_3$; green; G. 3,8: *Langite,* $\dot{C}u_4\ddot{S}\dot{H}_4$; blue; G. 3,5; COVELLITE, CuS; bluish-black; on coal in O. F. yields sulphur dioxide.

Section III. Alone in R. F. on coal, yield an arsenical odor, or a magnetic residue, or both.

Group I. Yield an arsenical odor and sometimes a magnetic residue also; fuse below 5.

1. *The fused mineral is dark and with successive portions of borax, on coal,* § 76, *d, shows cobalt or nickel persistently:*

ERYTHRITE, § 202. ANNABERGITE, § 236.

2. *Yield finally a residue (magnetic) which reacts strongly and persistently for iron with borax, giving a blackish to bottle-green glass on coal, becoming yellow in O. F. on wire,* § 76, *d:*

PITTICITE; $\ddot{F}e,\ddot{A}s,\ddot{S},\dot{H}$; a strong sulphur reaction with soda. PHARMACOSIDERITE, I; $\ddot{F}e_4\ddot{A}s_3(\ddot{P})\dot{H}_{15}$; H. 2,5. SCORODITE, IV; $\ddot{F}e\ddot{A}s\dot{H}_4$; H. 3,5–4. *Arseniosiderite,* $\dot{C}a_3\ddot{F}e_3\ddot{A}s_2\dot{H}_6$; yellowish-brown; fibrous; silky lustre.

3. *The residue is not magnetic:*

Trögerite, $\ddot{U}_3\ddot{A}s\dot{H}_{12}$; with S. Ph. shows uranium: *Chondrarsenite*; $\dot{M}n,\ddot{A}s,\dot{H}$; yellow to rdh-yellow grains; with S. Ph. shows manganese strongly: *Durangite,* $\dot{N}a,\ddot{A}l,\ddot{A}s(\ddot{F}e,\dot{L}i,\dot{M}n,F)$; orange to yellowish-red; gives fluorine reactions; *Pharmacolite,* $\dot{C}a_2\ddot{A}s\dot{H}_6$; colorless, gray, or sometimes reddish, from cobalt arsenate, and then gives a slightly blue bead with S. Ph.

Group II. Yield a magnetic residue but no arsenical odor.

1. *Yield a more or less distinctly green flame, either alone in the forceps, or moistened with H_2SO_4 in powder on platinum wire.*

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All except *Ludwigite* yield very decided reactions for phosphoric acid with magnesium, or with ammonium molybdate, § 109, *b*, *c*. Either of these tests may be used to confirm the presence of phosphoric acid in case of doubt.*

a. Alone, gives a red flame (lithium), perhaps with some green flame also: TRIPHYLLITE, $\dot{L}i(\dot{F}e,\dot{M}n)_2\ddot{P}$; fusible at 1.5 to 2.

b. With borax in O. F. on wire give a red, *manganese* bead (cold): TRIPLITE, $\dot{F}e_2\dot{M}n\ddot{P}+MnF_2$; brown to brnh-black; fuses easily; the borax bead in a pure O. F. finally becomes more or less pure amethyst-red; with potassium bisulphate a fluorine reaction: *Sarcopside*, flesh-red to lavender-blue, behaves similarly: *Hureaulite*, $\dot{M}n_5\ddot{P}_2\dot{H}_5(\dot{F}e)$, yields water and no fluorine reaction. *Childrenite*; $\dot{F}e,\dot{M}n,\ddot{A}l,\ddot{P},\dot{H}$; yields water; fuses only on the edges; the borax bead is reddish-yellow to yellowish-red.

c. The borax bead in O. F. on wire is yellow (iron) when cold: *Ludwigite*, $\dot{M}g_3\dot{F}e\ddot{F}e\ddot{B}$; fus. diff.; gives strong boric acid reactions; alone, may color the gas blowpipe flame green. The following six are fusible below 5. VIVIANITE, V; $\dot{F}e_3\ddot{P}\dot{H}_8$, bluish, blkh-green, white; **H.** 1,5–2; **G.** 2,6–2,7. DUFRENITE, IV; $\ddot{F}e_4\ddot{P}_2\dot{H}_5(\dot{F}e)$; leek-green to blkh-green, becoming brownish on exposure; **H.** 3,5–4; **G.** 3,2–3,4. *Borickite*; $\dot{C}a,\ddot{F}e,\ddot{P},\dot{H}$; amorphous; rdh-brown; **G.** 2,7. DIADOCHITE; $\ddot{F}e,\ddot{S},\ddot{P},\dot{H}$; brown to yellow; with soda a strong sulphur reaction; **G.** 2. *Cacoxenite*; $\ddot{F}e_2\ddot{P}\dot{H}_{12}(\ddot{A}l,F)$; ochre to lemon-yellow; very soft; **G.** 2,3. *Beraunite*, $\ddot{F}e_5\ddot{P}_3\dot{H}_{14}$; hyacinth-red to rdh-brown; **G.** 2,9–3. Compare *Limonite*, which shows considerable phosphoric acid only when impure, and may fuse somewhat; also *Childrenite*, above.

2. *Yield alone a strong lithium flame:* LEPIDOLITE (*lithia mica*), $(\dot{K},\dot{L}i)\ddot{A}l\ddot{S}i_3(\dot{M}n,\ddot{M}n)$; micaceous structure. Compare TRIPHYLLITE, above.

3. *Effervesce strongly with hot dilute HNO_3 or HCl, yielding carbon dioxide:*

SIDERITE, § 223: *Ankerite*; $\dot{C}a,\dot{M}g,\dot{F}e,(\dot{M}n),\ddot{C}$; resembles Siderite, but the HCl solution, boiled with a little nitric acid, treated with excess of ammonia and filtered, yields an abundant precipitate with ammonium oxalate; if this is filtered out a precipitate is also obtained on adding sodium phosphate: *Mesitite* is similar, but yields little or no precipitate with ammonium oxalate.

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* The nitric acid solution of the commoner minerals readily gives a decisive result with ammonium molybdate.

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4. *Soluble in HCl, leaving, after careful evaporation to dryness, little or no residue (silica) insoluble in HCl:**

a. Yield little or no sulphur reaction with soda: HEMATITE, § 218: TURGITE, p. 267: LIMONITE, § 219: GÖTHITE, III, $\dot{F}e\dot{H}$, reddish to blackish-brown, good clvg. in one direction, ignition-loss 10 %. Compare WOLFRAMITE, p. 267, which leaves, however, a partly yellow residue.

b. Yield a strong sulphur reaction with soda (compare *Hematite* and *Limonite*, above, which may show sulphur, owing to impurities): *Copiapite* ($\ddot{F}e,\ddot{S},\dot{H}$); in yellow, pearly scales and granular; G. 2,14: *Jarosite* ($\ddot{F}e,\ddot{S},\dot{H}$, also $\dot{K}$ or $\dot{N}a$); in small, yellow, tabular crystals; G. 3,25; also concretionary and earthy (*Gelbeisenerz*, G. 2,7—2,9): *Apatelite* ($\ddot{F}e,\ddot{S},\dot{H}$); $H_2O=3{,}96$ %. Compare *Pissophanite*.

5. *Gelatinize with HCl:*

a. Yield much water: *Cronstedtite*, $\dot{F}e_3\ddot{F}e\ddot{S}i_2\dot{H}_4(\dot{M}g,\dot{M}n)$; black, str. dark-green; swells and fuses on edges. *Nontronite*, (var. of *Chloropal*), straw-yellow to greenish; shines on rubbed surface; infus. Compare *Palagonite*, below.

b. Yield little or no water (compare ALLANITE, ILVAITE, *Fayalite*, and *Hortonolite*, p. 267; *Allochroite*, p. 277): *Knebelite*, $\dot{F}e\dot{M}n\ddot{S}i$, crystalline massive; gray, red, brown, green; strong manganese reactions.

6. *Decomposed by hydrochloric acid, leaving a residue of silica without gelatinizing:*†

a. Pyrosmalite, $\dot{F}e_4\dot{M}n_5\ddot{S}i_8\dot{H}_7FeCl_2$; gives a copper chloride flame with copper oxide.

b. Micaceous cleavage (and structure in part); yield little or no water: *Astrophyllite;* $\dot{K},\dot{N}a,\dot{F}e,\ddot{F}e,\dot{M}n,\dot{T}i,\ddot{S}i$; the strong HCl solution boiled with tin shows titanium; fusible. LEPIDOMELANE;

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* They must be tested in the finest possible powder, since some dissolve very slowly. All properly belonging here become *very magnetic* on coal in R. F. The careful student, only working for practice, may safely depend upon the non-formation of a silica skeleton with S.Ph., § 112, *a*, to distinguish between the minerals of this sub-division 4 and the *silicates* in the following ones, 5, 6, and 7; but then before beginning with the minerals in 4, above, WOLFRAMITE and CHROMITE, in 7, should be brought in here, since they also give no silica skeleton. The same may be true of *Emery*. See foot-note, just below.

† *Emery* (impure corundum, containing oxides of iron, usually magnetite) leaves a residue of impure alumina and may leave silica. Good emery scratches quartz very easily and scratches topaz; it is usually blackish-gray to nearly black when quite impure.

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K̇,Ḟe,F̈e,Ä̇l,S̈i(Ḣ) ; aggregate of minute, dark green to black scales; fusible.

c. Not included above :

a. Yield water : *Chalcodite ;* Ṁg,Ḟe,F̈e,Ä̇l,S̈i,Ḣ ; yellowish to greenish-bronze, velvety coatings on hematite and calcite ; brass-like to sub-metallic lustre ; fusible : *Palagonite ;* K̇,Ċa,Ṁg,F̈e,Ä̇l,S̈i, Ḣ ; sometimes gelatinizes ; amorphous ; brnh-yellow, str. same ; fusible : *Gillingite ;* Ḟe F̈e,S̈i,Ḣ ; compact, amorphous ; brownish-black ; fus. diff. : *Xylotile ;* Ṁg,Ḟe,F̈e,S̈i,Ḣ ; fibrous, asbestiform ; fus. diff. ; *Chloropal* (*pinguite*) ; Ḟe,F̈e,Ä̇l,S̈i,Ḣ ; greenish ; very soft ; slightly resinous lustre ; fus. diff. ; some varieties are only partially decomposed by HCl. Compare *Glauconite ; Stilpnomelane ;* (impure *Limonite, Hematite, clay iron ores,* leave a pulverulent residue of clay and silica ; **G. 3,** or more ; str. ochre-yellow to brnh-red).

b. Yield no water : *Allochroite* (*lime-iron garnet*), Ċa$_3$F̈eS̈i$_3$; greenish, brown, black ; fuses easily ; no clvg. ; may gelatinize imperfectly.

7. *Not attacked by HCl, or only slightly.*

a. Yield little or no water : CHROMITE, § 198. WOLFRAMITE, p. 267 : *Megabasite,* Ṁn Ẅ(Ḟe) ; str. yellowish ; **G. 6,45–6,94** ; behaves similarly ; gives a strong manganese reaction with borax.

Arfvedsonite, N̈aḞeF̈eS̈i$_8$, fuses easily with intumescence ; black ; str. greenish ; cleaves at 123°50′ : *Aegirine,* similar, cleaves at 93°. ALMANDITE (iron-alumina garnet), I, Ḟe$_3$Ä̇lS̈i$_3$; red ; **H. 7–7,5** ; fus. 3, quietly ; no clvg. : compare *Allochroite,* above. *Babingtonite,* (Ċa,Ṁn,Ḟe,F̈e,S̈i), fuses easily to a black glass ; fused with soda, dissolved in HCl, treated with excess of ammonia and filtered, the filtrate gives an abundant ppt. with ammonium oxalate : *Acmite,* N̈a$_2$ḞeF̈e$_2$S̈i$_4$, also fuses easily to a black glass, but yields little final ppt. with ammonium oxalate ; cleaves at about 93°.

Compare *Crocidolite,* below : also BIOTITE, p. 291, and TOURMALINE, EPIDOTE, AMPHIBOLE, PYROXENE, and HYPERSTHENE. p. 292 *et seq.*

b. Yield water : *Crocidolite* (N̈a,Ṁg,Ḟe,S̈i,Ḣ), lavender-blue, fibrous, fuses easily with intumescence : *Glauconite,* (K̇,N̈a,Ṁg,Ḟe, S̈i,Ḣ), green, earthy, soft, fus. 3, quietly.

Compare PROCHLORITE, p. 290 : CHLORITOID (Ṁg,Ḟe,Ä̇l,S̈i,Ḣ) is similar to it, but **H. 5–6.**

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Section IV. Minerals not included in the foregoing sections.

Sub-Section I. After long ignition alone react alkaline; hardness below 5.

1. ULEXITE, $\dot{N}aCa_2\ddot{B}_6\dot{H}_{18}$; *very* soft; fibrous; fuses easily; the dilute HCl solution shows boric acid with turmeric paper.

2. *Effervesce briskly (yielding carbon dioxide) with dilute nitric or hydrochloric acid, at least on heating, and leave little or no residue; streak colorless or pale.*

Gay-Lussite, $\dot{N}aCa\dot{C}\ddot{C}\dot{H}_5$; fuses easily; yields water. *Staffelite*; $\dot{C}a,\ddot{P},\ddot{C}(F,\dot{H})$; fus. 3; yields little water; phosphoric acid reactions. WITHERITE, IV, $\dot{B}a\ddot{C}$; gives a strong barium flame-coloration; fuses rather easily. BARYTOCALCITE, V; $\dot{B}a\dot{C}a\ddot{C}_2(\dot{M}n)$, fuses on thin edges; gives a barium flame; after ignition has usually a greenish color: *Bromlite*, IV, $(\dot{B}a,\dot{C}a,\ddot{C})$, similar reactions (according to Plattner is not green after ignition, but Dana quotes an analysis showing considerable manganese). STRONTIANITE, $\dot{S}r\ddot{C}$, generally sends out little projections in the forceps, B.B., but fuses only on thinnest edges; gives a strontium flame-coloration; the dilute HCl solution soon gives a ppt. on addition of a drop or two of dilute H_2SO_4: CALCITE,* III, $\dot{C}a\ddot{C}$, H. 3; rhombohedral clvg. perfect; infus.; effervesces briskly with cold dilute HCl or HNO_3: *Chalk* is a soft, earthy variety of Calcite: ARAGONITE, IV, $\dot{C}a\ddot{C}$, behaves like Calcite, but has a good clvg. in only one direction, if at all. DOLOMITE, $\dot{C}a\dot{M}g\ddot{C}_2$; H. 3,5—4; best distinguished from Calcite by little or no effervescence with cold dilute acids; the concentrated HCl solution yields a ppt. on addition of H_2SO_4; yields no water: MAGNESITE, $\dot{M}g\ddot{C}$; H. 3,5—4,5; resembles Dolomite, but the concentrated HCl solution gives no ppt. with H_2SO_4: *Hydromagnesite*, $\dot{M}g_4\ddot{C}_3\dot{H}_4$, is distinguished from compact Magnesite by yielding water: *Hydrodolomite* differs from Dolomite by yielding water: *Predazzite* and *Pencatite*, perhaps mixtures of Calcite and Brucite, resemble Hydrodolomite in behavior. Compare *Pyrochroite* and *Brucite*, p. 279, which may effervesce slightly, owing to impurities.

3. *Yield with soda a strong sulphur reaction; all fusible:*

GYPSUM. $\dot{C}a\ddot{S}\dot{H}_2$; H. 1,5—2; one perfect clvg. when well crystallized, giving thin plates; yields water; ANHYDRITE, $\dot{C}a\ddot{S}$; no

* Some of the following may blacken B.B., owing to the presence of manganese, but they do not give a decided manganese reaction with S.Ph. when dissolved only in rather small quantity; see RHODOCHROSITE, p. 281.

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water; the HCl solution, slightly diluted, gives an abundant ppt. with barium chloride: CELESTITE, $\dot{S}r\ddot{S}$, finally gives a good strontium flame-coloration: BARITE, $\dot{B}a\ddot{S}$, finally gives a distinct barium flame.

4. *Yield a fluorine reaction with potassium bisulphate; structure not micaceous; streak colorless or pale:*

FLUORITE, CaF_2, perfect octahedral clvg., fuses with some difficulty. CRYOLITE, VI, Na_3AlF_6; fuses very easily to a white enamel; intensely ignited and then treated with cobalt solution, gives a blue mass; H. 2,5: *Chiolite*, II, (Na,Al,F), H. 4, is a similar mineral, as are also *Arksutite* and *Chodneffite*, which yield no water, and *Pachnolite*, *Thomsenolite*, and *Gearksutite*, which yield water. Compare *Yttrocerite*.

5. *Not included above; dissolve easily in hydrochloric acid:*

BRUCITE, $\dot{M}g\dot{H}$; foliated, with perfect basal clvg.; also fibrous (*Nemalite*); H. 2,5; infus.; yields water; with cobalt solution shows magnesia. *Pyrochroite*, $\dot{M}n\dot{H}(\dot{M}g,\dot{C}a,\dot{C})$; also foliated; yields strong manganese reactions; Compare PSILOMELANE and WAD, p. 281.

6. *Not included above:* Some specimens of TALC and MUSCOVITE, p. 291.

Sub-Section II. Yield one of the following special reactions.

Group I. Yield a lithium flame; give no dark streak.

1. *Micaceous structure or aggregates of scales:*

LEPIDOLITE, p. 275; imperfectly decomposed by acids; quite fusible; yields little or no water: *Cookeite* ($\dot{K},\dot{L}i,\ddot{A}l,\ddot{S}i,\dot{H},F$); exfoliates remarkably; yields water; fuses on thin edges: *Cryophyllite* ($\dot{K},\dot{L}i,\dot{F}e,\ddot{F}e,\ddot{A}l,\ddot{S}i,F$); fuses easily; decomposed by HCl.

2. *Not micaceous; all fusible:*

AMBLYGONITE, $2\,\ddot{A}l\ddot{P}+3(Li,Na)F$; fuses at 2; the HNO_3 solution shows phosphoric acid, § 109, *c*; alone gives a yellowish-red flame: *Hebronite*, which is similar, gives a strong lithium flame: SPODUMENE, $\dot{L}i,\ddot{A}l\ddot{S}i_4(\dot{K},\dot{N}a)$; one perfect cleavage (pinacoidal) and sometimes a good prismatic cleavage at 87°; G. 3,1–3,2: PETALITE, $\dot{L}i\ddot{A}l\ddot{S}i_8(\dot{N}a)$; G. 2,4–2,5; usually easily distinguished from Spodumene by uneven fracture and generally by lack of cleavage, as well as by greasy to vitreous lustre; Spodumene has a more or less pearly lustre on cleavage face: *Castor* is a variety of Petalite.

Group II. Yield reactions for boric or phosphoric acid.

1. *Yield reactions for boric acid:*

a. Warwickite ($\dot{M}g,\dot{F}e,\ddot{T}i,\ddot{B}$); titanium reactions.

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b. DATOLITE, $\dot{C}a_2\ddot{B}\dot{S}i_2\dot{H}$; gelat. with HCl; alone, a green flame.

c. Dissolve in HCl without gelatinizing: *Sussexite* $(\dot{M}g,\dot{M}n)_2\ddot{B}\dot{H}$; fibrous; strong manganese reactions. *Boracite*, $2\dot{M}g_3\ddot{B}_4+MgCl_2$; H. 7 (crystals); shows chlorine, § 104, *a*, 1. *Hydroboracite*, $\dot{C}a\dot{M}g_2\ddot{B}_6\dot{H}_{12}$; H. 2; yields 26 % of water. *Szaibelyite*, $\dot{M}g\ddot{B}\dot{H}$; H. 3–4; yields 7 % of water. *Lüneburgite*, $\dot{M}g_3\ddot{P}\ddot{B}\dot{H}_8$; yields phosphoric acid reactions. *Larderellite* (*hydrous ammonium borate*); yields ammoniacal water. Compare *Danburite*, below.

d. Only slightly attacked by HCl: AXINITE, VI; $\dot{C}a_6Fe_2\ddot{A}l_3\ddot{B}\dot{S}i_{10}(\dot{M}n)$; fuses easily to a green or dark glass and alone colors the flame green. *Danburite* $(\dot{C}a,\ddot{B},\dot{S}i)$; H. 7; alone gives a green flame; no water: *Howlite*, similar in composition, but yielding water; H. 3,5 or less. Compare TOURMALINE, p. 285, which is pyroelectric.

2. *Yield phosphoric acid reactions:*

a. Fusible below 5: Compare AMBLYGONITE, Group I, 2, above: *Autunite*, $\ddot{U}_2\dot{C}a\ddot{P}\dot{H}_8$; fuses easily; yields water; with S. Ph. in R. F. a green bead: APATITE, $3\dot{C}a_3\ddot{P}+CaF_2(Cl)$; fuses at 4,5—5, quietly. *Wagnerite*, $\dot{M}g_3\ddot{P}+MgF_2$; fuses at 4; no water; soluble in dilute H_2SO_4. *Brushite*, $(\dot{C}a,\ddot{P},\dot{H})$; fuses easily; $H_2O=26$ %. *Isoclasite*, $\dot{C}a_3\ddot{P}+\dot{C}a\dot{H}+\dot{H}_4$; similar, but $H_2O=18$; H. 1,5; G. 2,9.

b. Infusible, or fusible above 5:

a. Yield water; become blue on ignition with cobalt solution:

WAVELLITE, $\ddot{A}l_3\ddot{P}_2\dot{H}_{12}(F)$: usually concretionary, with radiated structure; H. 3,2–4; G. 2,3; $H_2O=24$ to 28 %: *Evansite*, $\ddot{A}l_3\ddot{P}\dot{H}_{18}$, $H_2O=40$; *Fischerite*, $\ddot{A}l_2\ddot{P}\dot{H}_8$; H. 5; $H_2O=23$. *Peganite*, $\ddot{A}l_2\ddot{P}\dot{H}_6$; G. 2,5; $H_2O=23$. *Zepharovichite*, $\ddot{A}l\ddot{P}\dot{H}_6$; H. 5,5; $H_2O=27$ to 29. *Amphithalite*, $H_2O=12.5$, *Tavistockite*, $H_2O=12$; both containing $\dot{C}a$; and *Coeruleolactite*, $H_2O=21.2$, are similar. Compare LAZULITE, *Berlinite* and Trolleite, below.

b. Yield little or no water, but become blue with cobalt solution: LAZULITE, $(\dot{M}g,\dot{F}e)\ddot{A}l\ddot{P}\dot{H}$; azure-blue; B.B. swells, whitens and crumbles; insoluble in acids; G. 3,1: *Berlinite*, $\ddot{A}l_2\ddot{P}_2\dot{H}$; grayish to pale red: little affected by acids; G. 2,6; *Trolleite*, $\ddot{A}l_4\ddot{P}_3\dot{H}_3$, pale green; G. 3,1, behaves similarly.

c. Not included above: TURQUOIS, $\ddot{A}l_2\ddot{P}\dot{H}_5(\dot{C}u)$; blue to green; B.B. gives a green flame and becomes brown and glassy, but does not fuse; with S. Ph. and tin shows copper; yields water; G. 2,6–2,8: *Monazite*, V; $(\dot{C}e,\dot{L}a,\dot{T}h)_3\ddot{P}$; rdh, brnh; scarcely fusible; decomposed with difficulty by HCl; G. 4,9–5,3: *Xenotime*, II; $\dot{Y}_3\ddot{P}$ (Ce);

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generally light brown ; infus.; insoluble in HCl ; G. 4,5. Compare APATITE, p. 280 ; yields no water.

Group III. With borax in O. F. on wire give (after cooling) strong manganese reactions, very small quantities producing a decided amethyst-red color, larger ones an intense amethyst-red or even opaque bead.

1. *The S. Ph. bead in R. F. on wire is blue, reddish-brown, or red, and reduced beside tin on coal shows tungsten :*

Hübnerite, $\dot{M}n\ddot{W}$; str. yellowish-brown ; G. 7,14. Compare WOLFRAMITE, p. 267, and *Megabasite.*

2. *Yield no tungsten reaction ; the moderately charged S. Ph. bead, reduced on wire, is generally colorless, or nearly so, when cold :*

a. In powder with HCl evolve hydrogen sulphide : *Helvite,* ($\dot{M}n$, $\dot{F}e$,$\dot{B}e$,$\dot{S}i$,MnS); fus. diff.; gelat. with HCl. Compare ALABANDITE and Hauerite, p. 266.

b. Warmed with dilute HCl or HNO_3 yield carbon dioxide abundantly and leave little or no insoluble residue ; RHODOCHROSITE, $\dot{M}n\dot{C}$; rose-red, gray, brown ; H. 3,5—4,5 ; B.B. infus., but blackens and may become glazed on very thin edges; a bit half the size of a pin's head gives a decidedly violet-red color to a bead of S. Ph. of ordinary size, § 9, *a,* when cold (distinction from manganiferous calcite, etc.).

c. Powdered and warmed with strong HCl evolve chlorine (the yellowish gases escaping from the test tube first redden and then at least partially bleach blue litmus paper); the HCl solution evaporated to dryness leaves little or no insoluble residue of silica (or silicates), except in some specimens of Wad: WAD, $\dot{M}n$,$\ddot{M}n$,$\dot{H}$; usually very soft ; earthy ; brown to blackish ; yields water. Compare PYROLUSITE, § 229, MANGANITE, § 230 ; PSILOMELANE, p. 267 ; *Lithiophorite.*

d. Decomposed by HCl, leaving on evaporation a residue of insoluble silica : TEPHROITE, $\dot{M}n_2\dot{S}i(\dot{M}g)$; fus. 3.5 (to 6 in magnesian varieties) ; gelat. with HCl. *Klipsteinite,* $\dot{M}n_3\ddot{M}n_2\dot{S}i_3\dot{H}_4(\ddot{F}e)$; yields water ; with HCl evolves chlorine, leaving slimy silica ; fusible. Compare RHODONITE, below, which may be partly decomposed.

e. Slightly or not at all attacked by HCl :

a. Carpholite, $(\ddot{M}n,\ddot{A}l,\ddot{F}e)_2\ddot{S}i_3\ddot{H}_3$; silky lustre ; fus. with intumescence to brown glass : *Ardennite,* ($\dot{C}a$,$\dot{M}n$,$\ddot{A}l$,$\ddot{F}e$,$\ddot{V}$,$\ddot{A}s$,$\ddot{S}i$,$\ddot{H}$) ; fuses to black enamel.

b. Yield little or no water : SPESSARTITE (manganese-alumina

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garnet), I, $(Mn,\dot{F}e)_3\ddot{A}l\ddot{S}i_3$; brnh-red; **H. 7**; no clvg.; fuses quietly at 3: *Piedmontite,* $\dot{C}a_4(\ddot{A}l,\ddot{M}n,\ddot{F}e)_3\ddot{S}i_6$; red to rdh-black; fuses easily with intumescence. RHODONITE, VI, $\dot{M}n\ddot{S}i(\dot{M}g,\dot{C}a,\dot{F}e)$; red, brnh-red, black on exposed surfaces; crystals show distinct clvg. at 92°.55′; blackens and fuses quietly, or nearly so, at 3: *Bustamite* is a variety containing $\dot{C}a\ddot{C}$; *Fowlerite* a zinciferous variety. *Richterite,* a variety of Amphibole; and *Schefferite,* a variety of Pyroxene, give strong manganese reactions.

Group IV. Yield with S.Ph. on wire reactions as follows:

1. *In O. F. a blue or violet bead, becoming blue in R. F.; streak brown or black:* ASBOLITE, a cobaltiferous WAD, having similar reactions, see Wad, p. 281.

2. *In O. F. a yellow bead, unchanged in R. F.* ZARATITE, $\dot{N}i_3\ddot{C}\ddot{H}_6$; emerald-green; dissolves with effervescence in hot HCl; on coal yields metallic nickel: *Genthite,* $\dot{M}g_2\dot{N}i_2\ddot{S}i_3\ddot{H}_6$; green, yellowish; decomposed by HCl without effervescing.

3. *In O. F. a colorless, yellowish, reddish, or greenish bead (cold), which assumes a decided color in R. F., while the mineral yields one of the following special reactions:*

a. *Molybdite,* $\ddot{M}o$; molybdenum reaction, § 99, *f;* the S.Ph. bead in R. F. is green.

b. The S.Ph. bead in R. F. is blue, reddish-brown or red, and reduced with S.Ph. and tin on coal shows tungsten, § 100, *d:* SCHEELITE, $\dot{C}a\ddot{W}$; str. white; fuses at 5; **H. 4,5–5,5**; the S.Ph. bead in R. F. is blue: *Tungstite,* $\ddot{W}$; yellowish; soft, earthy; infus. Compare WOLFRAMITE, p. 267, and *Megabasite.*

c. The moderately charged S.Ph. bead becomes in R. F., at least with tin on coal, more or less distinctly violet when cold:

a. The following are more or less fusible [and not perfectly decomposed by HCl]. TITANITE, V. $\dot{C}a,\dot{T}i,\ddot{S}i(\dot{F}e,\dot{M}n)$; fuses at 3 to 4; **H. 5–5,5**; *Guarinite,* IV, is dimorphous with Titanite and behaves like it; **H. 6.**

b. Fusible; decomposed by HCl without gelatinizing: *Keilhauite,* V; $\dot{Y}\dot{C}a_4\ddot{A}l\dot{T}i_3\ddot{S}i_6$; **H. 6,5**; fuses with some bubbling: *Schorlomite,* I (also massive); $\dot{C}a_4\ddot{F}e\ddot{S}i_3\dot{T}i_4$; **H. 7–7,5**; decomposed with difficulty, yielding slimy silica: *Tscheffkinite,* $(\dot{C}a,\dot{C}e,\dot{F}e,\ddot{S}i,\dot{T}i)$; B.B. glows, swells much and finally fuses; with HCl yields gelatinous lumps of silica. *Mosandrite,* $(\ddot{K},\dot{N}a,\dot{M}g,\dot{C}a,\dot{C}e,\dot{L}a,\dot{D}i,\dot{T}i,\ddot{S}i,\ddot{H})$; **H. 4**; yields water; with HCl a dark red solution, yellowish on warming.

c. Infusible or nearly so: *Aeschynite,* $\dot{C}e_3\dot{T}h\dot{T}i\ddot{C}b(\dot{L}a,\dot{D}i,\dot{Y},\dot{C}a,$

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Fe); B.B. swells and becomes rdh-brown, but scarcely fuses; yields columbium reactions: RUTILE, II, $\dot{T}i(\ddot{F}e,\ddot{M}n)$; usually rdh-brown; str. yellowish-brown to nearly colorless; lustre more or less adamantine to metalloidal; in stout prismatic crystals, also acicular; infusible; G. 4,2–4,3: *Octahedrite* (Anatase), II, dimorphous with Rutile, resembles it in general, but the crystals, usually quite small, have pyramidal faces predominant and show perfect basal clvg.; G. 3,9: *Brookite*, III, usually in thin, tabular crystals, is another dimorphous form of $\dot{T}i$, similar in behavior; it includes the iron-black, sub-metallic *Arkansite*, usually showing better developed, thicker, pyramidal forms.

Group V. Yield a sulphur reaction with soda on coal.

1. *Gelatinize with HCl:*

Hauynite, much like Nosite in composition, but usually more $\dot{C}a$ and $\dot{K}$, fuses quietly at 4.5; generally blue; clvg. dodecahedral; *Nosite*, $3(\dot{N}a\ddot{A}l\ddot{S}i_2)+\dot{N}a\ddot{S}(\dot{C}a,Na,Cl)$; usually gray or brown; fuses quietly at 4.5: LAPIS LAZULI, similar composition; generally rich blue and massive-granular; fuses at 3: *Ittnerite*, similar composition, but yields $\dot{H}$ and fuses with intumescence; gray to dark bluish-gray: *Scolopsite*, massive, gryh-white to rdh-gray, similar in composition and behavior, but sometimes without water.

2. *Decomposed by HCl without gelatinizing, or are but slightly attacked; infusible or nearly so; become blue with cobalt solution (except some Pissophanite).*

Svanbergite ($\dot{K},\dot{N}a,\ddot{A}l\ddot{P},\ddot{S},\dot{H}$); yields phosphoric acid reactions: ALUMIAN, $\ddot{A}l\ddot{S}_2$, yields no water: ALUNITE, $\dot{K}\ddot{A}l_3\ddot{S}_4\dot{H}_6$; generally fine-grained, earthy, compact; H. 3,5–4; insoluble in HCl: ALUMINITE, $\ddot{A}l\ddot{S}\dot{H}_9$; dull white; very soft; easily soluble in HCl: *Felsobanyite*, $\ddot{A}l_2\ddot{S}\dot{H}_{10}$, is similar in behavior; G. 2,3; $H_2O=37$. *Pissophanite* ($\ddot{A}l,\ddot{F}e,\ddot{S},\dot{H}$); amorph., pitch-like, very easily crumbling; B.B. some varieties turn brown or black and with borax show much iron. *Zippeite*($\ddot{U},\ddot{S},\dot{H}$); uranium reactions.

[Sub-Section III. Minerals not included in the preceding Sub-Sections.

Note. Students using the tables only for practice will pass at once to Sub-Section IV. p. 291.

Group I. Fusibility from 1 to 5.

1. *Give a perfect jelly with hydrochloric acid:*

a. Yield water: Edingtonite ($\dot{B}a,\ddot{A}l,\ddot{S}i,\dot{H}$); dil. HCl solution yields ppt. with H_2SO_4; G. 2,7: NATROLITE, $\dot{N}a\ddot{A}l\ddot{S}i_3\dot{H}_2$; fus. 2, to a clear glass quietly; HCl solution treated with ammonia and

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filtered, gives little or no ppt. with ammonium carbonate; H_2O= 9%: Compare ANALCITE. *Scolecite*, $\dot{C}a\ddot{A}l\ddot{S}i_3\dot{H}_3$; fuses easily to a bubbly enamel; often curls up; H. 5,5; pyroelectric: *Laumontite*, $\dot{C}a\ddot{A}l\ddot{S}i_4\dot{H}_4$; fuses easily to white enamel, often curling up; H. 3: *Mesolite* and *Thomsonite* resemble Scolecite; not pyroelectric: *Phillipsite*, IV, $(\dot{K},\dot{C}a)\ddot{A}l\ddot{S}i_4\dot{H}_4$; fus. 3 with slight intumescence to white enamel; often crumbles; generally in cruciform twins: *Gismondite*, similar, but in apparent tetragonal pyramids. Compare APOPHYLLITE, *Okenite*, ANALCITE, in 2, *a*; they may gelatinize imperfectly.

b. Yield little or no water: *Sodalite*, I, $3(\dot{N}a\ddot{A}l\ddot{S}i_2)+NaCl$; H. 5,5 to 6; *Eudialyte*, III, $\dot{N}a_4\dot{C}a_2\dot{F}e\dot{Z}r_2\ddot{S}i_{12}$; both show chlorine: Sod. fuses to a clear, colorless glass; Eud. to an opaque pistache-green glass, and shows zirconium, § 67, *b*. WOLLASTONITE, $\dot{C}a\ddot{S}i$, fuses quietly to a colorless, semi-transparent glass; the HCl solution, freed from silica, yields little or no ppt. with ammonia, much with ammonium carbonate. Compare PECTOLITE, 2, *a*.

Nephelite, III $(\dot{N}a,\dot{K})\ddot{A}l\ddot{S}i_2$, (includes *Elaeolite*, with greasy lustre): *Meionite*, II, $\dot{C}a_6\ddot{A}l_4\ddot{S}i_9$; H. 5,5 to 6; *Melilite*, $(\dot{N}a,\dot{C}a,\dot{M}g,\ddot{A}l,\dot{F}e,\ddot{S}i)$; H. 5: the HCl solution of all, freed from silica, yields a ppt. with ammonia: Meionite fuses with intumescence and glowing to a bubbly glass: the solution of Neph. filtered from the ppt. caused by ammonia, yields little or no more with ammonium oxalate; that of Melilite yields much. Compare *Cancrinite*. *Barsowite*, $\dot{C}a_2\ddot{A}l_2\ddot{S}i_5$, behaves like Mel.; fus. 4, quietly. Compare *Gehlenite* and *Tachylite*.

2. *Decomposed by hydrochloric acid with separation of silica, but without gelatinizing perfectly:*

a. Yield water: APOPHYLLITE, $\dot{C}a\ddot{S}i\dot{H}_2$; PECTOLITE, $\dot{N}a\dot{C}a_4\ddot{S}i_6\dot{H}$; *Okenite*, $\dot{C}a\ddot{S}i_2\dot{H}_2$; ANALCITE, $\dot{N}a\ddot{A}l\ddot{S}i_4\dot{H}_2$: all easily decomposed, giving gelatinous lumps of silica; fresh Anal. often gelat. perfectly, while its HCl solution, freed from silica, yields a considerable ppt. with excess of ammonia. At first Anal. B.B. becomes cloudy, then fuses to a clear glass; no clvg.; H_2O=8%. Pect. fuses easily with slight bubbling, to a white, translucent, enamel-like glass; H_2O=4; gelat. after fusion. Apoph. and Okenite yield much H_2O; not easily decomposed after fusion; the former fuses at 1.5 to a bubbly, white glass; the latter at 2.5 to 3, with frothing, to a porcelain-like mass. Compare *Sepiolite* and *Xonaltite*.

The three following have H. 2,5 to 3: *Pyrosclerite*, $\dot{M}g_6\ddot{A}l\ddot{S}i_4\dot{H}_4$, green, clvg. in one direction, H_2O=11 %, fus. quietly at 4; *Choni-*

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crite ($\dot{M}g,\dot{C}a,\ddot{A}l,\check{S}i,\dot{H}$), whitish, no clvg., $H_2O=9$, fus. 3.5 to 4 with some bubbling; *Jollyte* ($\dot{M}g,\dot{F}e,\ddot{A}l,\check{S}i,\dot{H}$), amorph., brown, str. light green, $H_2O=13$, fus. diff. with some intumescence. *Jefferisite* and *Vermiculite*, resembling Pyrosclerite, swell extraordinarily B.B.

Catapleiite, $\dot{N}a\ddot{Z}rSi_3\dot{H}_2$; fus. 3, quietly to white enamel; shows zirconium, § 67, *b*: Compare *Mosandrite*. *Brewsterite*, $(\check{S}r,\dot{B}a)\ddot{A}l\check{S}i_6\dot{H}_5$; the dil. HCl solution gives a ppt. with H_2SO_4.

The four following intumesce more or less and fuse, with some curling up, to enamel-like masses: STILBITE, IV, $\dot{C}a\ddot{A}l\check{S}i_6\dot{H}_6$; HEULANDITE, V, $\dot{C}a\ddot{A}l\check{S}i_6\dot{H}_5$ (both with one perfect clvg.); CHABAZITE, rhombohedral, $\dot{C}a\ddot{A}l\check{S}i_4\dot{H}_6$; PREHNITE, $\dot{C}a_2\ddot{A}l\check{S}i_3\dot{H}$. Prehnite loses 4.3 % water on ignition; the others 15 to 20. *Mordenite*, related to Stilbite, fuses quietly; $H_2O=12$.

Sepiolite (*Meerschaum*) $\dot{M}g_2\check{S}i_3\dot{H}_4$; fus. on thin edges; absorbs water readily; $H_2O=10$ %. *Deweylite*, $\dot{M}g_4\check{S}i_3\dot{H}_6$, amorphous, lustre waxy, does not absorb water; $H_2O=20$. *Sordavalite* ($\dot{M}g,\dot{F}e,\ddot{A}l,\check{S}i,\dot{H}$) amorph., brnh-black, fus. 2.5 to a black glass; decomposed with difficulty by HCl; $H_2O=4$.

b. Yield little or no water: (Compare *Pectolite, Prehnite, Chonicrite*, above.) *Tachylite* ($\dot{N}a,\dot{C}a,\dot{M}g,\dot{F}e,\check{S}i$); amorph.; fus. 2,5 to a black glass quietly. WERNERITE, II, $\dot{N}a\dot{C}a_2\ddot{A}l_2\check{S}i_6$, fus. 2,5, with intumescence, to a white, bubbly glass, not easily rounded; rather good clvg. at 90°. *Paranthite*, *Ekebergite* and *Passauite* are related minerals. *Wöhlerite*, $\dot{N}a_6\dot{C}a_7\ddot{C}b\ddot{Z}r_3\check{S}i_9$; yellow, reddish, gray; fus. 3; shows columbium and zirconium reactions. LABRADORITE, $\dot{N}a\dot{C}a_3\ddot{A}l_4\check{S}i_{12}$; *Anorthite*, $\dot{C}a\ddot{A}l\check{S}i_2$; both fuse quietly, Lab. at 3.5; Anorth. at 4.5. The former has two unequal cleavages at 94°, is not entirely decomposed by HCl; usually gray, brown, green, and iridescent on cleavage face: Anorth. cleaves with nearly equal ease at 94°. 12′. GROSSULARITE, $\dot{C}a_3\ddot{A}l\check{S}i_3$, H. 7; largely decomposed by HCl; fus. 3, quietly; no clvg.

3. *Dissolves in hydrochloric acid without separation of silica: Struvite*, $(NH_4)_2MgPO_4+6H_2O$; heated with potassa solution, yields ammonia.

4. *Almost or entirely unaffected by acids.* (Compare *Pyrophyllite*, which B.B. may be rounded on edges.)

Harmotome, $\dot{B}a\ddot{A}l\check{S}i_5\dot{H}_5$; yields water; the partial HCl solution gives a ppt. with H_2SO_4. TOURMALINE ($\dot{K},\dot{N}a,\dot{L}i,\dot{M}g,\dot{M}n,\dot{F}e,\ddot{A}l,\dot{B},\check{S}i$); H. 7 to 7,5; boric acid reaction, § 113, *a*; easily fus. to infus.; usually quite pyroelectric. *Thermophyllite* ($\dot{M}g,\check{S}i,\dot{H}$); *Euphyllite*

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(Ċa,Ä̇l,S̈i,Ḣ); *Margarite* (Ċa,Ä̇l,S̈i,Ḣ); all micaceous: Therm. swells much; $H_2O=11$: Euph. (easily decomposed by H_2SO_4) and Marg. (with difficulty) yield little H_2O, 4 to 4,5 %. Compare MUSCOVITE, p. 287, and BIOTITE.

Gümbelite (K̇,Ä̇l,S̈i,Ḣ), short fibrous, swells in fan-like forms and fuses in slender fibres; $H_2O=7$; not attacked by HCl or H_2SO_4. *Leucophanite* (Ċa,B̈e,S̈i,NaF); H. 3,5 to 4; fus. 3, quietly to a clear colorless glass; one excellent clvg. direction; phosphoresces reddish-violet when heated or struck. *Wilsonite* (K̇,Ċa,Ä̇l,S̈i,Ḣ); H. 3; swells and fuses at 2, to a whitish glass; yields H_2O; distinct clvg. at 90°. *Nohlite* (C̈b,Ü̇,Ẏ,Ḣ); blkh-brown, lustre vitreous; fus. diff.; yields some H_2O; otherwise like Samarskite, p. 268. Compare *Sordavalite*, 2, *a*.

DIALLAGE (Ċa,Ṁg)S̈i; fus. 3.5; metalloidal-pearly lustre; one distinct clvg. direction. AUGITE (Ċa,Ṁg,Ḟe)S̈i; DIOPSIDE (Ċa,Ṁg)S̈i: both varieties of *Pyroxene;* distinct prismatic clvg. at about 93°; fus. 3.5 to 4, quietly, or nearly so; Aug. to a dark, Diop. to a whitish glass; Aug. is dark-colored, green or black; Diop. is light green, gray or colorless.

HORNBLENDE, green to black, and TREMOLITE, white or light gray and greenish, are varieties of *Amphibole*, resembling Pyroxene varieties in behavior and composition, but cleaving at 124° 30′: ACTINOLITE, in bladed crystals, belongs here, as well as *Asbestus* and *Amianthus* (in part): *Nephrite*, fracture splintery; somewhat greasy to the touch; H. 6; greenish; related to Tremolite.

ORTHOCLASE, $\dot{K}\ddot{A}lSi_6$ and ALBITE, $\dot{N}a\ddot{A}lSi_6$, fuse quietly; Orth. at 5, Alb. at 4; they are not attacked by HCl: the former cleaves very distinctly in two planes at 90°; the latter has two cleavages at 93° 30′, the planes being finely striated. OLIGOCLASE $(\dot{K},\dot{N}a,Ċa)_2\ddot{A}l_2S̈i_3$, resembles Albite, but fus. 3.5: *Hyalophane* resembles Orth. but fused with soda, treated with dilute HCl and filtered, yields a ppt. with H_2SO_4 (barium).

EPIDOTE, $Ċa_4(\ddot{A}l,\ddot{F}e)_3S̈i_6$; H. 6,5; fus. 3 to 3.5, with intumescence to a dark, bubbly mass; gelat. after fusion; two rather distinct cleavages at 115°; usually dark yellowish-green: *Zoisite* is similar, but gives B.B. a light-colored mass and cleaves well in one direction; light colors.

The three following have H. 6,5 to 7,5: GROSSULARITE (alumina-lime garnet) $Ċa_3\ddot{A}lS̈i_3$, fus. 3; and VESUVIANITE, $Ċa_3(\ddot{A}l,\ddot{F}e)_2S̈i_7$, fus. 3, with intumescence; both gelat. after fusion: *Pyrope* (precious garnet), fus. 4,5, blood-red: Vesuv. cleaves somewhat distinctly

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at 90° ; the others have no clvg.: Gross. is in part strongly attacked by HCl. *Monzonite* (Na,Ca,Fe,Al,Si) resembles compact Gross., but does not gelatinize after fusion ; not decomposed by HCl or H_2SO_4. Compare BERYL, p. 290 ; also *Euclase, Iolite, Biotite.*

Obsidian, Pitchstone (greasy lustre), *Pearlstone* and *Pumice* are volcanic glasses, amorphous and often containing a little water ; fus. 3.5 to 4, with some swelling, to bubbly, white glasses or a porcelain-like mass. Obsid. has vitreous lustre and large, conchoidal, sharp-edged fracture ; Pumice has a froth-like, very porous structure.

Group II. Infusible or fusibility above 5.

1. *Assume a fine blue color when ignited with cobalt solution.*

a. Yield water : *Gibbsite,* AlH_3, H. 2,5–3,5, $H_2O=34.5$ %, soluble in potassa solution. The three following cleave easily in one direction : *Diaspore,* AlH, H. 6, $H_2O=15$; *Pholerite,* $AlSi_2H_2$, H. 1, $H_2O=15$, found in pearly scales, *Xanthophyllite* (Ca,Mg,Al,Si,H) $H_2O=4.5$.

The four following yield gelatinous silica with HCl : *Allophane,* $AlSiH_5$, amorph., H. 3, gelat. perfectly, usually colors the flame green (copper), $H_2O=42$ % ; *Halloysite,* $AlSi_2H_4$, earthy, H. 1–2, $H_2O=16$; *Samoite,* $Al_2Si_3H_{10}$, H. 4, $H_2O=30$; lamellar; *Collyrite,* earthy, Al_2SiH_{10}, H. 1–2, $H_2O=33.5$.

The following are soft, amorphous, and attacked with difficulty by HCl ; $H_2O=12$ to 16 % : *Cimolite,* $Al_2Si_2H_6$, somewhat tough ; *Porcelain Clay* (var. of *Kaolinite*) $AlSi_2H_2$, fine-grained and earthy ; when pure forms minute pearly scales. Here belong ordinary *Clays,* plastic with water and having a clay-like odor ; also some *Lithomarge,* $H_2O=14$; *Schrötterite,* $H_2O=35$; *Miloschite* and *Bole,* $H_2O=24$ to 26 ; they are not plastic when wet ; the last two crumble with a slight crackling noise in water. Compare *Pyrophyllite, Fibrolite, Seybertite* and *Agalmatolite,* in *b* ; also *Ripidolite.*

b. Yield little or no water : *Pyrophyllite,* $AlSi_4H_2$, clvg. perfect in one direction, exfoliates prodigiously B.B., $H_2O=5$ %, H. 1–2; *Westanite,* brick-red, is related to it. *Agalmatolite,* (var. *Pinite*) $KAlSiH$, H. 2–3. MUSCOVITE, V, $KAl_2Si_4(H)$; perfect clvg. into thin, tough, elastic plates; H. 2,5; not attacked by acids. *Brandisite* (Mg,Ca,Al,Si,H), H. 4–5 ; cleaves in one direction ; decomposed by H_2SO_4. ANDALUSITE, AlSi ; H. 7, 5 ; clvg. distinct at about 91° ; G. 3,2 ; and CYANITE, AlSi, H. 6 or less ; clvg. at 106°, one clvg. plane being especially distinct ; usually bluish ; G. 3, 6; both are little attacked by acids. *Fibrolite,* G. 3, resembles Cyanite.

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Chiastolite, H. 5,5 or less, is altered Andalusite, generally found in the shape of four prismatic crystals, grown together in parallel directions and having the space between them filled in with clay slate.

The four following are not attacked by acids. TOPAZ, $5(\ddot{A}l\ddot{S}i)+Al_2SiF_{10}$, H. 8, B.B. remains transparent, but may change from yellow to rose-red; excellent basal clvg, G. 3,5, occasionally pyro-electric. *Rubellite* (*Lithia Tourmaline*), H. 6,5, no clvg., G. 3, B.B. whitens and often swells to a slaggy mass; pyro-electric. CORUNDUM (*Sapphire, Ruby*) $\ddot{A}l$, H. 9, G. 4; and CHRYSOBERYL, $\dot{B}e\ddot{A}l$, H. 8,5, G. 3,7, are slowly soluble in S. Ph.

Compare LEUCITE, 3, *b*. QUARTZ, 4, *b*, may become pale reddish-blue with cobalt solution.

2. *Entirely or almost entirely soluble in hydrochloric (or nitric acid) without gelatinizing or leaving any considerable residue of silica.* (Compare URANINITE, p. 268.)

Hydrotalcite ($\dot{M}g,\ddot{A}l,\dot{C},\dot{H}$); yields much water; dissolves with slight effervescence; with cobalt solution gives a faint rose color. *Parisite* ($\dot{C}e,\dot{L}a,\dot{C},Ca,F$); slowly soluble in HCl, with effervescence; the not too acid solution yields with oxalic acid a white ppt., becoming brick-red on ignition. *Polycrase* ($\dot{Y},\dot{C}e,\dot{F}e,\dot{Z}r,\dot{T}i,\ddot{C}b$) gives reactions for zirconium, columbium and titanium.

Fluocerite (Ce,F), yellow, G. 4,7–4,9; and *Yttrocerite* (Ca,Ce,Y,F), G. 3,5; give fluorine reactions: *Bastnäsite* ($\dot{C}e,\dot{L}a,\dot{C},Ce,F$), similar, but yielding carbon dioxide.

3. *Decomposed by HCl, either gelatinizing or leaving a residue of silica:*

a. Yield water: *Uranotile* ($\dot{C}a,\dot{U},\ddot{S}i,\dot{H}$); acicular, lemon-yellow crystals, blackening B.B.; yields uranium reactions. *Xonaltite,* $\dot{C}a_4\ddot{S}i_4\dot{H}$; infus.; the HCl solution, freed from silica, treated with ammonia and filtered, yields a heavy ppt. with ammonium oxalate. *Thorite,* $\dot{T}h\ddot{S}i\dot{H}_2$, black, str. dark brown, and *Cerite,* $\dot{C}e_2\ddot{S}i_2\dot{H}$, rdh-gray, have G. 4,7–5 and gelat. with HCl. The slightly acid solution of Cerite yields with oxalic acid a white ppt,, becoming rdh-brown on ignition.

Nontronite, $\dot{F}e\ddot{S}i_3\dot{H}_6$, amorph.; yellowish-green; its powder is blackened by cold potassa solution. *Xylotile* and *Gillingite,* p. 277. *Wolchonskoite* ($\ddot{A}l,\ddot{C}r,\ddot{S}i,\dot{H}$), amorph., dark leek-green, gives chromium reactions. *Sepiolite,* $\dot{M}g_2\ddot{S}i_3\dot{H}$; B.B. whitens and shrinks; with HCl a gelatinous mass; $H_2O=10\,\%$. *Bastite* ($\dot{M}g,\ddot{S}i,\dot{H}$) and *Chrysotile,* $\dot{M}g_3\ddot{S}i_2\dot{H}_2$ show metalloidal lustre, Bast. on one clvg.

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surface, Chrys. on its grayish to yellowish-green, silky masses: both are decomposed by HCl, more easily by H_2SO_4, without gelatinizing; B.B. Bast. becomes brown, Chrys. white; $H_2O=12$ % in both. *Metaxite,* lustre feeble, silky; compact and fine fibrous; similar to Chrysotile. *Cerolite,* $\dot{M}g_4\ddot{A}l\check{S}i_5\dot{H}_{15}$; amorph., H. 2–3, $H_2O=30$; with cobalt solution a pale flesh color.

SERPENTINE, $\dot{M}g_3\check{S}i_2\dot{H}_2$; decomposed by HCl without gelat.; usually massive, compact; feeble sub-resinous, greasy or waxy lustre; H. 3–5,5; $H_2O=12$ to 13 %. The following behave similarly, but show crystalline structure and clvg.: *Picrophyll,* H. 2,5, $H_2O=10.5$; *Picrosmine,* H. 2,7, $H_2O=9$; *Marmolite,* H. 2,5, $H_2O=15.7$; all greenish: *Kämmererite,* III, H. 1,5–2, $H_2O=13$, is red; *Kotschubeite,* optically biaxial, otherwise resembling Käm. Compare PROCHLORITE and RIPIDOLITE, decomposed with difficulty by HCl; also *Deweylite.*

Antigorite, thin lamellar, with one perfect clvg., H. 2,5; *Monradite,* crystalline foliated, H. 6; and *Neolite,* very soft, with soapy feel, contain $\dot{M}g,\dot{F}e,\check{S}i,\dot{H}$; they all, as well as *Clintonite* ($\dot{M}g,\dot{C}a,\ddot{A}l,\check{S}i,\dot{H}$), metalloidal pearly lustre, one perfect clvg. direction, are decomposed by HCl without gelat. and have $H_2O=4$ to 6 %.

b. Yield little or no water (compare the last minerals of *a*): *Gadolinite* ($\dot{Y},\dot{C}e,\dot{B}e,\dot{F}e,\check{S}i$), gelat. perfectly; G. 4–4,3; B.B. swells and often glows in a peculiar way, finally becoming rounded on thin edges; black to blkh-green. *Gehlenite,* $\dot{C}a_3\ddot{A}l\check{S}i_2$, gryh-white, G. 3; rounded B.B. on thin edges. CHRYSOLITE (*Olivine*) $\dot{M}g_2\check{S}i$, generally olive-green, H. 7, gelat. CHONDRODITE ($\dot{M}g,\check{S}i,Mg,F$), generally yellowish to brownish; gelat.; yields fluorine reactions. *Monticellite* (Batrachite) containing also $\dot{C}a$, yields, after addition of ammonia and filtering, a ppt. with ammonium oxalate. *Forsterite* (Boltonite) $\dot{M}g_2\check{S}i$, one distinct clvg. direction, gives slimy silica; G. 3,2. *Leucite,* $\dot{K}\ddot{A}l\check{S}i_4$; no clvg.; usually in trapezohedral crystals; decomposed without gelat.; often becomes blue with cobalt solution; G. 2,5; H. 5,5.

4. *Minerals not included above; they are classified by hardness:*

a. Hardness below 7 (the hardness of quartz): BIOTITE $(\dot{K},\dot{M}g)_3(\ddot{A}l,\ddot{F}e)\check{S}i_3$, optically uniaxial, decomposed by H_2SO_4; MUSCOVITE, $\dot{K}\ddot{A}l_2\check{S}i_4(\dot{H})$; and TALC, $\dot{M}g_3\check{S}i_4\dot{H}$, feel soapy, laminae inelastic; all three yield little or no water, and cleave perfectly into thin laminae. *Soapstone* is compact Talc. Compare *Pyrophyllite. Margarodite* and PHLOGOPITE, both biaxial, are decomposed by H_2SO_4. Near Muscovite are *Margarite* (decided pearly lustre) and

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Oellacherite; after fusion with soda the HCl solution of Oell. yields a ppt. (barium) with H_2SO_4.

PROCHLORITE, often in minutely foliated, granular masses, and RIPIDOLITE cleave perfectly into thin, inelastic laminae; they, as well as *Delessite* (short-fibrous) contain Mg, Fe, Al, Si, H; yield 12 % water and have H. 2,5, or less. Del. is easily decomposed by HCl; the others slowly, but more readily by H_2SO_4. Rip. whitens B.B., fusing on the edges to a grayish-yellow enamel; Prochl. blackens and becomes slightly magnetic. *Leuchtenbergite,* yellow, is uniaxial; Rip. is biaxial. *Penninite,* uniaxial, is a related mineral, dark green. CHLORITOID is also similar to the foregoing in composition; H. 5–6; $H_2O=7.5$.

Cerolite, 3, *a,* is mostly decomposed by HCl. *Beauxite* (Al, Fe, H), amorph.; gryh-white, rdh-brown; H. 3; little attacked by acids; $H_2O=20$. Compare *Clays.*

BRONZITE, a variety of *Enstatite,* (Mg, Fe)Si; H. 5–5,5; one perfect clvg. direction, with marked metalloidal-pearly lustre: *Anthophyllite,* Mg_3FeSi_4, is similar, but has lustre less decided and cleaves at 124°.30′: HYPERSTHENE, closely related to Bronz. cleaves distinctly at 86°.30′.

Aeschynite (Ce, Y, Zr, Fe, Cb, Ti), black, str. light brownish, and *Euxenite* (Ce, Y, U, Cb, Ta, Ti), brnh-black, str. rdh-brown (B.B. unchanged), yield columbium reactions and have metalloidal-greasy lustre: Aesch. fused with potassium bisulphate and treated with water, leaves a residue which, dissolved in HCl and diluted, colors turmeric paper orange-yellow (Brush); B.B. swells. *Pyrochlore* (from Miask), I, brnh-red, str. pale yellow, is similar in reactions, but shows no zirconium.

OPAL (Si, H); infus., may yield little or no water; H. 6–6,5; G. 2–2,3; amorph.; nearly or entirely soluble in boiling potassa solution, which with ammonium chloride afterwards yields a gelatinous silica ppt.

Compare *Orthoclase* and *Hyalophane.*

b. Hardness 7 or more (compare *Opal,* above): QUARTZ, III, Si (includes *Amethyst, Agate, Jasper, Hornstone, Flint, Chalcedony;*) B.B. unaltered; with enough soda fuses to a clear bead. *Tridymite* is a dimorphous form of Si, in microscopic, hexagonal scales. IOLITE, $Mg_2Al_2Si_5$; H. 7; G. 2,6; bluish to grayish; fus. 5—5,5. STAUROLITE (Mg, Fe)Al_2Si_2; G. 3,6; brown to brnh-red; infus.

The four following have H. 7,5; BERYL (*Emerald, Aquamarine*) III, Be_3AlSi_6, B.B. becomes cloudy-white and rounded on thinnest

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edges, basal clvg. distinct: *Euclase*, V, $\dot{B}e_2\ddot{A}l\check{S}i_2\dot{H}$; B.B. similar; clvg. perfect at 90°: *Phenacite*, $\dot{B}e_2\check{S}i$, G. 3, and ZIRCON, II, $\dot{Z}r\check{S}i$, G. 4,4–4,6, are infusible; the latter loses its color B.B., and yields a zirconium reaction, § 67, *b*.

TOPAZ, IV ($\ddot{A}l,\check{S}i,Al,F$); distinct basal clvg.; H. 8; G. 3,5; shows fluorine, § 107, *b*. *Ouvarovite*, $\check{C}a_3\ddot{C}r\check{S}i_3$; emerald-green; infus., but darkens transiently; with borax shows chromium; H. 7,5–8; G. 3,5. SPINEL, I, $\dot{M}g\ddot{A}l$, including the very dark, ferruginous *Ceylonite* (*pleonast*); H. 8; G. 3,5–3,6; B.B. unaltered, or only darkens transiently; slowly soluble in S.Ph. GAHNITE (*Zinc Spinel*) gives a zinc coating with soda and borax; it includes *Automolite* ($\dot{Z}n,\dot{M}g)\ddot{A}l$, *Kreittonite* ($\dot{M}g,\dot{F}e,\dot{Z}n)(\ddot{A}l,\ddot{F}e)$, and *Dysluite* ($\dot{F}e,\dot{M}n,\dot{Z}n)(\ddot{A}l,\ddot{F}e)$.

Diamond, nearly pure carbon; H. 10, scratching corundum; G. 3, 5—3,6 for crystals, 3–3,4 for massive varieties.]

Sub-Section IV. Minerals not included in Sub-Sections I and II.

This Sub-Section is intended only for practice; all of the minerals in it will be found also in Sub-Section III, to which references are given simply for a statement of the composition of the minerals.

A. STRUCTURE EMINENTLY MICACEOUS.

TALC (*foliated variety*), p. 289; laminae flexible, but inelastic; greasy or soapy feel; H. 1–1,5; yields little or no water; fus. diff. on thin edges, and may exfoliate somewhat; in the forceps with cobalt solution becomes pale flesh-red. PYROPHYLLITE (*foliated variety*), p. 287; laminae flexible, but inelastic; H. 1; B.B. curls and swells extraordinarily, to many times its original volume; whitens and fuses on thin edges; with cobalt solution assumes a fine and pure blue color; yields more or less water: VERMICULITE, p. 285; also swells most remarkably; fuses with some difficulty to a grayish-black mass; yields water.

MUSCOVITE, p. 289; laminae elastic and tough; H. 2–2,5; yields little or no water; B.B. fuses on thin edges; shows potassium, p. 93, foot-note; little affected by H_2SO_4; usually light colored: PHLOGOPITE, p. 289, resembles Muscovite in general, but is decomposed by H_2SO_4; its laminae are sometimes inelastic, owing to incipient decomposition; usually yellowish-brown to brownish-red, and often with a metalloidal-pearly lustre on cleavage surfaces: BIOTITE, p. 289, also resembles Muscovite, but is decomposed by H_2SO_4 and in very rare cases may show little or no potassium; it is generally quite dark and usually gives a decided iron reaction with borax.

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RIPIDOLITE, PENNINITE and PROCHLORITE, p. 290, have flexible laminae, generally inelastic and never very elastic; **H. 2,5** or less; fus. diff., Prochlorite becoming more or less magnetic in R. F.; all yield much water; are decomposed by H_2SO_4; show no potassium, p. 93, foot-note: they are scarcely to be distinguished from one another with certainty except by chemical analysis and optical examination.

B. STRUCTURE NOT MICACEOUS.

Group I. Yield little or no water.

1. *Fusibility not above* 5:

a. Gelatinize perfectly with HCl, but some only slowly and in finest powder:

SODALITE, p. 284, shows chlorine, § 104, *a*, 1: WOLLASTONITE, p. 284; the HCl solution freed from silica (see foot-note, p. 295) yields little or no ppt. with ammonia, but much with ammonium carbonate; (compare PECTOLITE, p. 295, which may gelatinize imperfectly and yield little water, but always some, and fuses at 2): NEPHELITE, p. 284; the HCl solution, freed from silica, yields a decided ppt. with ammonia.

b. Do not gelatinize perfectly with hydrochloric acid:

a. Yields a boric acid reaction with Turner's flux; TOURMALINE, p. 285.

b. The following fuse with decided intumescence and also gelatinize after fusion: WERNERITE, II, p. 285; fuses to a whitish, bubbly glass; **G. 2,6–2,8**; prismatic clvg.: ZOISITE, V, p. 286; fuses to a white, pale yellowish, or light gray slag; **G. 3–3,3**; light colored, with one excellent cleavage direction: (compare PREHNITE, p. 295, which always yields water, although but about 4 %, and may have one distinct cleavage direction, but has **G. 2,8–2,95**): EPIDOTE, V, p. 286; usually pistache-green or brownish-green; fuses to a dark mass, often magnetic in R. F.; **G. 3,25–3,5**; generally shows one excellent cleavage direction, and sometimes another at 115° 24′ with the former: VESUVIANITE, II, p. 286, usually brown, is further distinguished from Epidote by general lack of good cleavage; does not become magnetic in R. F.; **G. 3,35–3,45.**

c. The following fuse for the most part quietly: GARNET, I, pp. 277 and 286; rarely shows any cleavage; **H. 6,5–7,5**; **G. 3,15** or more; gelat. after fusion, more or less completely (compare PECTOLITE, p. 295, usually with fibrous, or slender columnar structure, and always yielding some water; **H. 5.**)

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AMPHIBOLE, p. 286 ; V ; crystals have perfect cleavage at 124°,30′; often in slender, bladed crystals and fibrous, or radiated masses; fus. 3,5–4, sometimes becoming magnetic in R. F.; **H. 5–6; G. 2,9–3,4:** PYROXENE, p. 286, **G. 3,2–3,5,** resembles Amphibole in fusibility and hardness, but has prismatic cleavage at 87°,5′, if any (sometimes has good cleavage parallel to the pinacoids) and is rarely fibrous in structure ; it is most surely distinguished from the FELDSPARS, below (apart from the cleavage) by **G. 3,2–3,5,** and especially from ORTHOCLASE by generally fusing at or below 4, as well as by showing no potassium (except a few rather rare varieties); compare HYPERSTHENE, p. 294, which fuses with much difficulty to a magnetic slag, and also shows no potassium.

FELDSPARS : these are characterized by two very good, but unequally easy cleavages, at about 85° to 90°, cleavage in one direction being very perfect ; there may also be a third, much less distinct cleavage direction ; they have **G. 2,5–2,8,** nearly, and **H. 6–7;** here are included—ORTHOCLASE, (*Common Feldspar*), V, p. 286 ; two good cleavages at 90°, one being very perfect ; fuses with some difficulty, at 4,5 to 5 ; always shows potassium, p. 93, foot-note ; not much decomposed by HCl, if at all : ALBITE, VI, and OLIGOCLASE, VI, p. 286, cleave at 93°,30′ to 94°, one cleavage surface usually showing fine, parallel twinning striations ;* Albite fuses at 4, OLIGOCLASE at 3,5 ; both may show potassium, and they are little affected by HCl, if at all ; they cannot be distinguished from one another except by chemical analysis and optical examination : LABRADORITE, VI, p. 285 ; fus. 3 ; differs from the two foregoing feldspars in being slowly, but very largely, decomposed by HCl in very fine powder, without gelatinizing ; usually dark gray to brown, with a fine play of color on one cleavage surface.

Compare IOLITE, p. 294, which shows no potassium and is partly attacked by HCl ; fus. 5,5.

2. *Fusibility above* 5:

a. Gelatinize perfectly with hydrochloric acid : CHONDRODITE, p. 289, yields a fluorine reaction with S.Ph., § 107, *b*. CHRYSOLITE, p. 289.

b. Do not gelatinize perfectly with hydrochloric acid :

a. With cobalt solution afford a fine, bright blue color (hard minerals must be finely powdered): PYROPHYLLITE (massive vari-

* *Microcline* is a triclinic potash-feldspar, cleaving at 90°,16′, and sometimes easily distinguished by its twinning striations from Orthoclase, which it closely resembles ; it includes the green *Amazon-stone*, easily losing color B.B.

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ety), p. 287; **H.** 3, or less. TOURMALINE (some light colored varieties), p. 285; shows boric acid with Turner's flux, § 113, *a*, and whitens B.B., but fus. diff. on thin edges, if at all.

(The five following minerals do not properly belong under a, since they do not give a fine bright blue color, but they may assume an impure blue tint: QUARTZ, see b, yields at most only a pale purplish or bluish-red color; OPAL, p. 296, may behave similarly: BERYL, see b, may yield a dark or grayish-blue mass, quite glassy if highly heated; ORTHOCLASE, p. 293, and IOLITE, see b, yield a mass which, if blue, is dark and decidedly fused).

The remaining minerals are all infusible: TOPAZ, p. 288, shows fluorine with S. Ph., § 107, *b*: LEUCITE, 289, is decomposed by HCl without gelatinizing: ANDALUSITE, p. 287, **H.** 7–7,5; **G.** 3,2; yields with S. Ph. a silica skeleton, even when in rather fine powder; sometimes shows good cleavage at 91°, 30′: CYANITE, p. 287, behaving similarly, has one perfect cleavage direction and another less perfect, at about 106° with the first; **G.** 3,5–3,7; **H.** 5–7: CORUNDUM, p. 288, dissolves slowly, but perfectly, in S. Ph.; cleavage rhombohedral, if any; **G.** 3,9–4; **H.** 9 (compare SPINEL, p. 295): CHRYSOBERYL, p. 288, behaves much like Corundum; **H.** 8,5; **G.** 3,65–3,8; may cleave distinctly in one direction, with another less distinct cleavage at right angles to the first.

b. Do not give a fine blue color with cobalt solution: URANINITE, p. 268: TALC (*Soapstone*) compact variety; **H.** 2,5 or less; greasy feel: TOURMALINE, p. 285; some varieties are nearly infusible; gives a boric acid reaction with Turner's flux, § 113, *a*: HYPERSTHENE (*Paulite*) p. 290; fus. diff. on edges to a dark slag, becoming magnetic in R. F.; dark colored and often with metalloidal lustre.

ZIRCON, p. 291; infus.; reaction for zirconium, § 67, *b*, after fusion with soda: ORTHOCLASE, p. 293; fus. 4,5—5 (and therefore not strictly belonging here): BERYL, p. 290; B.B. becomes enamel-like (whitish) and rounded on thin edges: IOLITE, p. 290; fus. 5.5; only one distinct cleavage direction, if any; **G.** 2,6–2,7; usually quite dichroic, being blue in one direction, by transmitted light, and grayish in another: ENSTATITE (*Bronzite*), p. 290; fus. diff. on thin edges; often fibrous structure; prismatic cleavage at 87°; lustre pearly or silky (and sometimes metalloidal) on cleavage surface: QUARTZ, p. 290; **H.** 7; **G.** 2,6–2,8; infus.; well fused with about one and one-half volumes of soda, gives a bead remaining clear when cold (compare OPAL, p. 296).

STAUROLITE, p. 290; infus., or nearly so; **H.** 7–7,5; with one

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and one-half volumes of soda does not fuse to a clear (cold) bead; after long ignition with the soda it gelatinizes with HCl: SPINEL, p. 291; infus.; H. 8; does not fuse to a clear bead with the soda, nor gelatinize afterward with HCl; (fused with one and one-half parts each of soda and borax on coal in O. F., the powdered mass boiled with HCl and a little HNO_3, the unfiltered solution slightly diluted, heated with a slight excess of ammonia and then filtered, yields a solution in which sodium phosphate produces an abundant ppt., especially after warming; distinction from Corundum).

Group II. Yield water.

1. *Fusibility not above 5*:

a. Gelatinize perfectly with hydrochloric acid:

NATROLITE, p. 283; IV; fus. 2, quietly to a clear glass; the HCl solution heated with ammonia and filtered yields little or no precipitate with ammonium carbonate (compare ANALCITE, below). SCOLECITE, p. 284; H. 5,5; fuses easily, to a white, bubbly enamel, generally curling up: LAUMONTITE, p. 284, H. 3,5–4, behaves similarly.

Compare PECTOLITE and APOPHYLLITE, below; and SEPIOLITE, p. 296, which fuses with difficulty.

b. Do not gelatinize perfectly with HCl (compare SEPIOLITE, p. 296; fus. diff.):

HARMOTOME, p. 285; the HCl solution, somewhat largely diluted and filtered, quickly becomes turbid on addition of dilute H_2SO_4.

The HCl solution of the two following, freed from silica,* yields little or no ppt. with ammonia: PECTOLITE, p. 284; fuses easily, sometimes with slight bubbling, to a white enamel; yields comparatively little water; gelat. perfectly after fusion, imperfectly before: APOPHYLLITE, p. 284; fuses easily to a white, bubbly glass and gives a potassium flame-coloration; gives slimy silica; after fusion is attacked with difficulty by HCl.

The HCl solution of the four following, freed from silica, yields a considerable ppt. with ammonia: ANALCITE, p. 284; I; trapezohedral forms predominant; never fibrous; fuses easily and quietly; gelat. imperfectly (sometimes perfectly): the three following fuse with more or less intumescence to enamels, or enamel-like glasses: PREHNITE, p. 285; H. 6–7; G. 2,8–3; yields little water: CHABAZITE, p. 285; H. 4–5; lustre vitreous; clvg. rhombohedral, not very

* The silica is removed by evaporating the solution to dryness, warming the residue with a few drops of HCl, then adding water and filtering out the now insoluble silica.

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perfect: STILBITE, p. 285; one perfect cleavage direction, with pearly lustre on cleavage surface.

2. *Fusibility above 5* :

a. Gelatinize perfectly with HCl : CERITE, p. 288 ; H. 5,5 ; G. 4,7—5 ; infus., but may become glazed B.B.: ALLOPHANE, p. 287 ; H. 3 ; amorphous ; ignited with cobalt solution, becomes blue : SEPIOLITE, p. 288 ; amorphous ; soft ; absorbs water readily ; fus. diff., or almost infus.; with cobalt solution becomes pale red or flesh color; does not always gelatinize perfectly.

b. Do not gelatinize perfectly with HCl (compare SEPIOLITE, above): KAOLINITE, p. 287; in microscopic, pearly scales ; also clay-like; very soft; with cobalt solution becomes fine blue : GIBBSITE, p. 287; generally stalactitic or mamillary; H. 2,5—3,5 ; infus.; with cobalt solution becomes fine blue ; rather easily soluble in potassa solution: OPAL, p. 290; amorphous ; vitreous to resinous, or waxy lustre; H. 5,5—6,5 ; infus., but often becomes cloudy B.B.; well fused with about one and one-half volumes of soda, gives a clear bead when cold; usually yields water ; with cobalt solution becomes, at most, reddish-blue.

Compare IOLITE, p. 294; fus. 5,5; often yields considerable water, through incipient and perhaps not very apparent alteration.

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SYMBOLS AND ATOMIC WEIGHTS OF THE ELEMENTS.

(Fownes' Chemistry.)

ELEMENTS.	SYMBOLS.	AT. WT.	ELEMENTS.	SYMBOLS.	AT. WT.
Aluminium	Al	27.4	Manganese	Mn	55.
Antimony	Sb	120.	Mercury	Hg	200.
Arsenic	As	75.	Molybdenum	Mo	96.
Barium	Ba	137.	Nickel	Ni	58.8
Bismuth	Bi	210.	Nitrogen	N	14.
Boron	B	11.	Osmium	Os	199.2
Bromine	Br	80.	Oxygen	O	16.
Cadmium	Cd	112.	Palladium	Pd	106.6
Cæsium	Cs	133.	Phosphorus	P	31.
Calcium	Ca	40.	Platinum	Pt	197.4
Carbon	C	12.	Potassium	K	39.1
Cerium*	Ce	92.	Rhodium	Rh	104.4
Chlorine	Cl	35.5	Rubidium	Rb	85.4
Chromium	Cr	52.2	Ruthenium	Ru	104.4
Cobalt	Co	58.8	Selenium	Se	79.4
Columbium (Niobium)	Cb (Nb)	94.	Silicium	Si	28.
			Silver	Ag	108.
Copper	Cu	63.4	Sodium	Na	23.
Didymium*	Di	95.	Strontium	Sr	87.6
Erbium*	E	112.6	Sulphur	S	32.
Fluorine	F	19.	Tantalum	Ta	182.
Gallium	Ga	68.?	Tellurium	Te	128.
Glucinum (Beryllium)	Be	9.4	Thallium	Tl	204.
Gold	Au	197.	Thorium	Th	235.
Hydrogen	H	1.	Tin	Sn	118.
Indium	In	113.4	Titanium	Ti	50.
Iodine	I	127.	Tungsten	W	184.
Iridium	Ir	198.	Uranium	U	240.
Iron	Fe	56.	Vanadium	V	51.2
Lanthanum*	La	93.6	Yttrium*	Y	61.7
Lead	Pb	207.	Zinc	Zn	65.2
Lithium	Li	7.	Zirconium	Zr	89.6
Magnesium	Mg	24.			

* Regarded as dyad.

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GENERAL INDEX.

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